



# **Extending the Season for Concrete Construction and Repair**

Phase I—Establishing the Technology

Charles J. Korhonen, Peter M. Semen, and Lynette A. Barna

February 2004

20040621 030

#### REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

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5e. TAS	DJECT NUMBER
	K NUMBER
5f. WOR	RK UNIT NUMBER
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) 8. PERF	ORMING ORGANIZATION REPORT
U.S. Army Engineer Research and Development Center Cold Regions Research and Engineering Laboratory 72 Lyme Road Hanover, NH 03755-1290  ERDC	C/CRREL TR-04-2
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)  10. SPO	DNSOR / MONITOR'S ACRONYM(S)
111-1-1	DNSOR / MONITOR'S REPORT MBER(S)

#### 12. DISTRIBUTION / AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited.

Available from NTIS, Springfield, Virginia 22161.

#### 13. SUPPLEMENTARY NOTES

#### 14. ABSTRACT

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15. SUBJECT TERMS	Antifreeze Cold weather co	ncrete	Freeze-thaw durability Freezing point		
16. SECURITY CLAS	SIFICATION OF:		17. LIMITATION OF OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			19b. TELEPHONE NUMBER (include area code)
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Phase I—Establishing the Technology

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Prepared for FHWA Pooled-Fund Program Project TPF-5(003)

#### **ABSTRACT**

The benefit of combining several commercial admixtures into a formulation to protect fresh concrete against freezing and promote significant strength development while the internal temperature of the concrete is below freezing was investigated. Laboratory studies developed eight potential admixture combinations for use at low temperatures. Each combination was shown in the laboratory to produce concrete that had reasonable workability, that could be entrained with air, that did not freeze until its internal temperature dropped to -5°C, that developed strength while held at -5°C as rapidly as did normal concrete held at 5°C, that could be entrained with air, and that could be finished at -5°C almost as rapidly as normal concrete held at 5°C. Five field trials were conducted to demonstrate that it is possible to mix, transport, place, finish, and cure concrete made with these admixture combinations at air temperatures as low as -20°C, with little or no thermal protection. Several batching sequences were demonstrated in the field trials to accommodate various haul distances and working times. The sequence where all admixtures were dosed into the truck at the jobsite created the most time for the concrete workers to place the concrete. Cost-wise, the admixture combinations tested in the field were shown to be less expensive than the conventional approach to winter concreting, which relies on heated shelters to keep the concrete warm while it cures. No special tools, techniques, or precautions were required to work with the concretes made with these admixtures.

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### **CONVERSION FACTORS**

Symbol	When You Know	Multiply By	To Find	Symbol
		Length		
mm	millimeters	3.93701 x 10 <sup>-2</sup>	inches	in
cm	centimeters	3.93701 x 10 <sup>-1</sup>	inches	in
m	meters	3.28084	feet	ft
m	meters	1.09361	yards	yd
km	kilometers	6.21371 x 10 <sup>-1</sup>	miles (statute)	mi
		Area		
mm <sup>2</sup>	square millimeters	1.55000 x 10 <sup>-3</sup>	square inches	in <sup>2</sup>
m²	square meters	$1.07639 \times 10^{1}$	square feet	ft²
m²	square meters	1.19599	square yards	yd <sup>2</sup>
•		Volume		
mL	milliliters	3.38140 x 10 <sup>-2</sup>	fluid ounces	fl oz
L	liters	2.64172 x 10 <sup>-1</sup>	gallons	gal
m³	cubic meters	$3.53147 \times 10^{1}$	cubic feet	ft <sup>3</sup>
m³	cubic meters	1.30795	cubic yards	yd <sup>3</sup>
		Mass		
kg	kilograms	2.20462	pound-mass, avoirdupois (avdp)	lbm
g	grams	3.52740 x 10 <sup>-2</sup>	ounces (avdp)	OZ
		Density		
kg / m³	kilograms per cubic meter	1.68555	pound-mass (avdp) per cubic yard	lbm / yd <sup>3</sup>
kg / m³	kilograms per cubic meter	6.24280 x 10 <sup>-2</sup>	pound-mass (avdp) per cubic foot	lbm / ft <sup>3</sup>
		Temperature (ex	ract)	
°C	degrees Centigrade	1.8 x (°C) + 32	degrees Fahrenheit	°F
		Pressure or Str	ess	
MPa	megapascals	1.45038 x 10 <sup>2</sup>	pound-force per square inch	psi
	Concr	ete Admixture Do	osage Rates	
mL / 100 kg	milliliters of admixture per 100 kg of cement	1.53378 x 10 <sup>-2</sup>	fluid ounces of admixture per 100 lbm of cement	fl oz / cwt
mL / m <sup>3</sup>	milliliters of admixture per cubic meter of concrete	2.58527 x 10 <sup>-2</sup>	fluid ounces of admixture per cubic yard of concrete	fl oz / yd <sup>3</sup>
L/m³	liters of admixture per cubic meter of concrete	2.01974 x 10 <sup>-1</sup>	gallons of admixture per cubic yard of concrete	gal / yd³

#### **PREFACE**

This report was prepared by Charles J. Korhonen, Peter M. Semen, and Lynette A. Barna, Research Civil Engineers of the Civil Engineering and Infrastructure Branch, U.S. Army Engineer Research and Development Center (ERDC), Cold Regions Research and Engineering Laboratory (CRREL), Hanover, New Hampshire. The work was funded by the FHWA pooled-fund program project TPF-5(003). The authors wish to acknowledge the ten state departments of transportation that used SP&R funds to support this work: Idaho, Michigan, Montana, New Hampshire, New York, Pennsylvania, Utah, Vermont, Wisconsin and Wyoming. In addition, the following are acknowledged for their contributions to this project: W.R. Grace and Master Builders donated the admixtures; Carroll Concrete (NH), Persons Concrete (NH), and Musson Concrete (WI) batched the concrete for field tests; and Howard Barker (PCA), Peter Keene (CRREL) and Bruce Lyndes (CRREL) took some of the photos.

Marcia Simon (FHWA), and the following state DoT representatives Chuck Corliss (NH), Peter Kemp (WI), Craig Graham (VT), Earl McArthur (MT), and Roger Apple (PA) technically reviewed this report.

The Commander and Executive Director of the Engineer Research and Development Center is Colonel James R. Rowan, EN. The Director is Dr. James R. Houston.

## Extending the Season for Concrete Construction and Repair

#### Phase I—Establishing the Technology

CHARLES J. KORHONEN, PETER M. SEMEN, LYNETTE A. BARNA

#### 1 INTRODUCTION

#### **Background**

When the weather turns cold, freshly placed concrete sets up more slowly, takes longer to finish, and gains strength less rapidly. To offset these problems, fresh concrete should never cool below 5°C for sections thicker than 1800 mm or below 13°C for sections thinner than 300 mm (ACI 1988). If concrete can be mixed and protected so that its temperature can be maintained at or above these levels, construction can stay on schedule and freezing will not be a problem. At air temperatures near 5°C and insulation, together with the heat generated inside the concrete, usually are sufficient to keep the concrete warm and the project on schedule. As the air temperature drops below 5°C, more elaborate protection, such as heated enclosures, becomes necessary. Should the weather get unexpectedly cold to freeze the concrete at an early age, the damage done by the 9% volume expansion of water turning into ice can destroy the concrete.

The U.S. Army Engineer Research and Development Center, Cold Regions Research and Engineering Laboratory (CRREL) has developed several formulations of antifreeze concrete that allow appreciable strength to be gained while the internal temperature of the concrete is below 0°C. To date, research has led to the development of two commercial prototype formulations for use at concrete temperatures down to -5°C at a Corps project in northern Michigan (Korhonen et al. 1997, Korhonen and Brook 1996); at the Tennessee Valley Authority, which used ordinary admixtures at -8°C (Korhonen et al. 1998); and the evaluation of over 50 expedient chemicals for use down to -10°C and lower by the Army in emergency situations (Korhonen 1999). CRREL proposed to extend this technology to common practice in a cooperative study, supported by state departments of transportation. This proposal was funded as FHWA project TPF-5(003).

#### The Problem

Currently, there are no commercially available admixtures, when used alone, that will prevent fresh concrete from freezing at an internal temperature of -5°C. Admixtures are available that allow concrete to gain strength at air temperatures below zero, but these admixtures, when used at their recommended dosages, will not prevent freezing. They promote strength gain by accelerating cement hydration, which sufficiently increases the rate of internally generated heat to maintain concrete temperatures above freezing until enough strength is developed to resist damage from freezing.

#### The Goal

The goal was to develop an antifreeze admixture formulation from existing commercial off-the-shelf admixtures. The resulting admixture was to prevent fresh concrete from freezing down to an internal concrete temperature of -5°C and to allow the concrete to gain appreciable strength while at that temperature. This work was to develop the tools to design, mix, place, and cure concrete in below-freezing weather.

With the relaxation of the concrete placing and curing temperature limits brought by an antifreeze concrete technology, a significant extension of the construction season should be economically feasible and convenient. With the successful development of a robust antifreeze concrete technology, we conservatively estimate a potential 10-million-cubic-meter market for winter concrete in the U.S. to support highway and street construction projects alone. The U.S. placed more than 100 million cubic meters of pavement during the summer of 1999 (Suprenant and Malisch 1999). Figure 1 illustrates that 3 to 4 more months of construction season would be available across the continental U.S. if the acceptable temperature for concrete work were  $-5^{\circ}$ C, instead of  $5^{\circ}$ C.

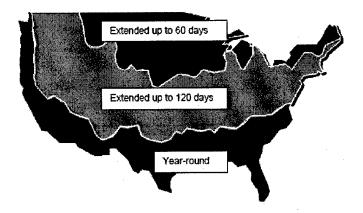


Figure 1. Extension of concrete construction season when allowable concrete temperatures are -5°C.

#### Emerging Technology

Experience in Europe (Gavrish et al. 1974) shows that chemical admixtures can depress the freezing point in fresh concrete and accelerate curing. An appropriate balance of such admixtures can permit placement of concrete without thermal protection in the form of heat or insulation at significantly lower temperatures than allowed by ACI standards.

Experience in the United States confirms that both proprietary (Korhonen and Brook 1996) and commercially available chemical admixtures (Korhonen et al. 1998) can create concrete of excellent quality when emplaced, unprotected, in subfreezing conditions. The following describe these two examples, respectively:

In the winter of 1994, several 550-  $\times$  610-  $\times$  15-cm concrete slabs were cast and cured unprotected at ambient temperatures that dipped below  $-15^{\circ}$ C in a repair of a horizontal surface at Soo Locks (Fig. 2). Each slab of concrete incorporated a different proprietary antifreeze admixture. In yearly visual inspections since 1994, each slab has proven to be of excellent quality after placement and as durable as any high-quality concrete.

In 1997, the Tennessee Valley Authority required repair of a concrete floor in an ice condenser room in a nuclear power plant (Fig. 3). To avoid shutting down the plant at a cost of nearly \$3M per day, the repair had to be performed at temperatures of -8°C. In this instance, combinations of conventional chemical admixtures were used to protect the concrete against freezing.



Figure 2. Unprotected antifreeze-admixture concrete, immediately after being finished and before being covered with plastic, at Soo Locks, Sault Ste. Marie, Michigan.



Figure 3. Low-temperature repairs were made to concrete floors in this nuclear power plant with the help of off-the-shelf admixtures.

#### Methodology

#### The Solution

The solution was to evaluate combinations of commercially available admixtures for their ability to depress the freezing point of water and to accelerate the hydration rate of cement. Using commercial products assures that they have been thoroughly tested for their effects on concrete and on compatibility among combinations of admixtures. Previous research (Korhonen and Brook 1996, Korhonen and Orchino 2001) showed that no single admixture, when used within recommended dosages, could provide enough freeze protection to meet the goal of this project, an internal concrete temperature as low as -5°C. Because standard practice places no limit on the number of admixtures that may be used in concrete, just on individual amounts, several were combined to produce the desired antifreeze effect. The admixtures chosen for this study had to meet the requirements of ASTM C 494 (1999a) or they had to be commercial products otherwise accepted by industry practice. The admixtures were first evaluated under controlled laboratory conditions to find the correct combination of admixtures that would not degrade freezeresistance, accelerate curing, and ensure workability at below-freezing temperatures.

#### Approach

Work began in October 2000 with the *Planning* phase of the study (Fig. 4). The concentration here was to research admixture product lines from commercial manufacturers, select individual admixtures to include in the program, and begin to formulate combinations (suites) of admixtures that might provide antifreeze capabilities. DOT study partici-

pants were solicited for input regarding typical concrete mixture designs employed in their highway applications. Using this feedback helped in selecting a single, standard mixture to base laboratory testing upon, so it could easily be transferred to field use later on. In this round, standard test methods were verified (and sometimes modified) for use at low-temperatures. In several instances entirely new test protocols were developed for measuring performance criteria particular to concrete containing low-temperature admixtures.

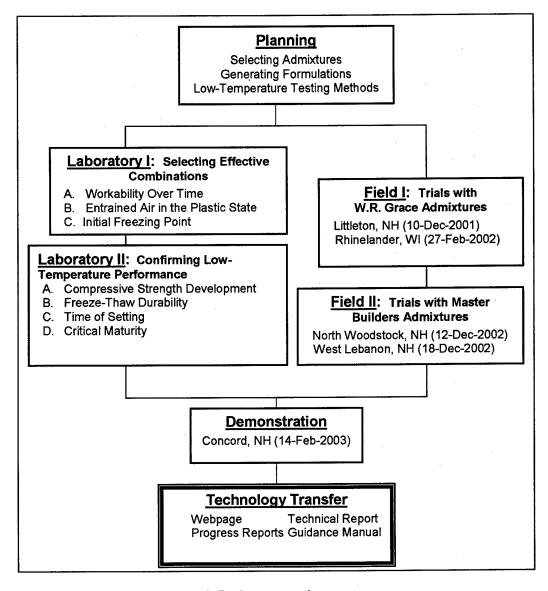


Figure 4. Project execution sequence.

Laboratory studies were quickly initiated in late 2000, using the project's first year to focus on developing antifreeze formulations from W.R. Grace & Co. products. The Laboratory I phase was used to screen for the best performing combinations of admixtures in the three critical areas of workability, entrained air content, and freezing point depression. Once we found formulations that met the initial criteria, we initiated confirmatory tests in the Laboratory II phase. Low-temperature performance was established in subsequent tests of strength, durability, setting time, and additional protection beyond the freezing point.

By September 2001, the end of the project's first year, we had developed four suites from W.R. Grace products and were ready for the *Field I* phase of full-scale trials with our DOT partners. In December 2001 and February 2002, we took two of the admixture combinations developed in the lab and used them at highway construction sites in New Hampshire and Wisconsin. At this point, halfway through the project's 3-year duration, we had successfully demonstrated our off-the-shelf admixture combinations under field conditions.

With more experience in formulating effective antifreeze suites under our belts, we returned to the laboratory to create suites using products from Master Builders, Inc. Between March 2002 and October 2002, we produced four more successful combinations with the new product line, repeating the *Laboratory I* and *Laboratory II* phases. Also, during this period, the W.R. Grace suites were optimized and their *Laboratory II* tests were completed to ensure that they were well-suited for highway application.

One of the newest formulations, utilizing the Master Builders products, was successfully field-tested at two sites in New Hampshire in December 2002 during the *Field II* phase. The feasibility of transferring the developed technology to our DOT partners was tested in the *Demonstration* phase during a record cold spell in February 2003 at Concord, New Hampshire—with excellent results. With two and one-half years of the project complete, we were able to hand off the oversight of mixture design, admixture dosing, concrete placement, and proper curing of antifreeze concrete to clients.

From March through September 2003, remaining Laboratory II work was completed and the focus turned to Technology Transfer. Providing progress reports to participants and posting our field results to the project webpage took place throughout the entire study. To wrap up, we concentrated on producing this technical report and developing a users guide to antifreeze concrete in the field.

#### Performance Requirements

As stated in the *Background* section, current industry guidance only allows concrete to be placed in cold weather when its internal temperature can be maintained at 5°C or higher. Typically, this is achieved with a combination of tenting and heating, insulation,

and heated raw materials. We wanted to eliminate the need for these expensive methods by using chemical admixtures to extend this lower temperature limit, allowing internal concrete temperatures as low as -5°C. The objective was to develop combinations of commercially available, currently approved, and commonly used admixtures that could be used to produce cold weather concrete that behaves like regular concrete at the time of placement and appearing "seamless" to the laborer at the end of the concrete chute. The cold weather concrete was required to meet the following performance requirements:

- Uses each admixture within manufacturer's published dosage range.
- Has an initial set time at -4°C\* that is no more than regular concrete at 5°C.
- Ensures "workable" concrete<sup>†</sup>:
  - Allows up to 45 minute transit time to a jobsite.
  - Allows at least 20 to 30 minutes of working time for emplacement and finishing after any additions, adjustments, or compliance testing at the jobsite.
- Protects fresh concrete from freezing down to at least -5°C.
- Develops compressive strength when cured at -4°C as well as or better than regular concrete cured at 5°C.
- Provides additional protection below the design temperature of  $-5^{\circ}$ C.
- Does not adversely affect long-term durability of concrete.
- Is compatible with steel reinforcement (i.e., non-corrosive).
- Does not promote alkali-silica reactions (ASR).
- Produces concrete able to accept air entrainment.
- Does not adversely affect finishability.
- Does not present significant problems with equipment use and cleanup in cold weather.

Effects of the admixture combinations on corrosion and ASR were not tested directly in this program. Only admixtures that were already known not to cause these problems were used. Because corrosion and ASR problems are chemically based, the offending reactions cannot proceed without the presence of the necessary reactants or catalysts. Conversely, testing on the long-term freeze—thaw durability of concrete with the admixture combinations and its ability to accept entrained air was conducted. The off-the-shelf

<sup>\*</sup> Set time and strength gain tests were performed on samples cured at -4°C, just above the target freezing point of -5°C, to ensure that results came from unfrozen specimens.

<sup>&</sup>lt;sup>†</sup> A full discussion of workability (slump) over time in antifreeze concrete follows in the *Laboratory I* section.

admixtures individually met standard specifications that check for these effects. However, we were concerned with the cumulative effects that combinations might have—even though combining admixtures without prior testing in this area is common practice today. The concern was that durability and air entrainment issues are not only chemically based, but involve physical processes as well that might be affected by admixture interactions. In addition, these problem areas are of specific interest in the cold regions.

#### Project Scope

Testing of the admixture combinations developed in this project was limited to concrete using ASTM C 150 (2002a) Type I or Type II portland cements. Other types of portland cements, non-portland cements, and blended portland cements containing slag, fly ash, silica fume, etc., were not explored; though the compatibility of these with anti-freeze admixtures should be addressed in future studies. Generally, we added our anti-freeze suites to concretes with a cement factor of 392 kg/m³, or 7 sacks. However, in the Rhinelander, Wisconsin, field testing, two suites were successfully adapted for use with a 476 kg/m³ cement factor (8½ sacks) to provide enhanced early age strength gain. All laboratory testing was performed on a locally available concrete mixture containing a water-cement (w/c) ratio of 0.44. This mixture was then modified with the admixtures chosen for this study.

#### 2 LABORATORY INVESTIGATION

#### **Materials**

#### Admixtures (General)

The admixture categories chosen for this study are shown in Table 1. The admixtures either met the requirements of ASTM C 494 (1999a), ASTM C 260 (2001a), or were commercial products otherwise accepted by industry practice. Admixtures, such as these, are used every day to produce concrete with enhanced or special properties. Each of these off-the-shelf products serves a particular function in creating an improved material performance. Our concern was how, when combined with one another, they would perform in cold weather.

Specification standard Description Type A Water-reducing Type B Retarding **ASTM** Type C Accelerating C 494 Water-reducing and retarding Type D (1999a) Water reducing and accelerating Type E Type F High-range water-reducing High-range water-reducing and retarding Type G Air-entraining ASTM C 260 (2001a) Corrosion-inhibiting (None) (None) Shrinkage-reducing

Table 1. Standardized concrete admixtures.

The three objectives of combining the admixtures in Table 1 into an antifreeze suite were to:

- Depress the freezing point of the water in the concrete to:
  - Protect from the damaging expansive pressures of ice formation.
  - Maintain liquid water for the hydration process.
- Accelerate the strength gain rate of concrete at low temperatures.
- Reduce the amount of water in concrete that requires protection from freezing.

As mentioned above, each admixture serves a specific role in industry today. A brief discussion of each category of admixture is provided below.

Water-Reducing/High-Range Water-Reducing (HRWR) Admixtures—ASTM C 494 (1999a) Type A, F. Commonly known as "plasticizers," these products are used to lower the w/c ratio in concrete, while maintaining workability. Conversely, they can also be used to raise the workability of a concrete mixture, while holding the w/c ratio constant. Type A water reducers typically decrease water demand by at least 5%, while Type F high-range water reducers cut water demand by more than 12%—sometimes more than 25%. Mid-range water reducers, for which there is no ASTM category, reduce water demand between these two limits. The downside of water reducers is that they can delay both setting times and early age strengths. The benefit of plasticizers in this study was that they reduced the amount of mixing water that needed to be protected from freezing.

As will be discussed later, combining moderate doses of prolonged-acting mid-range plasticizers with small doses of shorter-lived high-range plasticizers gave the best performance in terms of workability and freeze protection. Once the effectiveness of the high-range admixture faded, the mid-range admixture continued to maintain good workability. We had the most success using polycarboxylate-based plasticizers in combination with the other admixtures used in our suites. However, this does not suggest that other plasticizer combinations would not perform well.

Retarding Admixtures—ASTM C 494 (1999a) Type B. These slow down cement hydration, and are regularly used for offsetting early stiffening and setting at high temperatures. In this study, they were used in an attempt to moderate early stiffening arising from high doses of chemicals that accelerate hydration. By themselves, retarding admixtures, because they are used in small dosages, have minimal effects on depressing freezing points.

Some mixture designs evaluated small to moderate doses of retarder to test their effectiveness at restraining accelerated mixtures. Unfortunately, the retarders did little to assuage early stiffing problems, but they did delay strength development. This may have some implications for placing bridge deck concrete, where it's best that strength does not begin to develop until the entire deck is placed, but retarders were not considered useful in this project where early age strength gain was essential.

Accelerating/Water Reducing and Accelerating Admixtures—ASTM C 494 (1999a) Type C, E. Normally used to speed up cement hydration, accelerators provide faster-setting concrete with enhanced early strength development. Although this is helpful for cold conditions, accelerating admixtures by themselves do not provide sufficient antifreeze performance because they are not typically dosed at high enough levels to provide meaningful freezing point depression.

Testing focused on non-chloride accelerators based on calcium nitrate and calcium nitrite. These chemicals do not promote corrosion of steel reinforcement and even provide protection against corrosion. Some accelerators tested were classified as Type E, offering the benefit of additional workability owing to their plasticizing effects.

Water-Reducing and Retarding/High-Range Water-Reducing and Retarding Admixtures—ASTM C 494 (1999a) Type D, G. These admixtures combine the set-retarding function of Type B with the plasticizing effects of Types A and F above. Excessive set retarding, especially in conjunction with low temperatures, can produce mixtures that set and gain strength very slowly. As discussed for retarders, delayed strength development can be beneficial for bridge deck construction, but for this study strength delay was seen as a problem. To avoid problems, we chose to control these effects independently of each other, administering the Type A, B, and F admixtures separately. Some of the retarding admixtures used in creating the antifreeze suites were classified as both B and D. However, at the low end of the dosage range that was employed, the admixtures performed as Type B products.

Air-Entraining Admixtures (AEA)—ASTM C 260 (2001a). Added in very small amounts, these admixtures create a matrix of tiny, well-spaced air bubbles in concrete that serve to make it more durable to repeated cycles of freezing and thawing. The air-entraining admixtures had no measurable effect on the freezing point of the concrete.

Acceptable air contents were achieved with air-entraining admixtures based on both salts of wood resins (vinsol) and fatty acid salts. Though not studied in detail, the vinsol resins seemed to produce entrained air most easily. In either case, controlling air contents to a specified level in the antifreeze mixtures was sometimes a challenge. High doses of admixtures, like accelerators, corrosion inhibitors, and some shrinkage reducers, tended to be harsh on air content. Conversely, some plasticizers and other shrinkage reducers worked synergistically with the air-entraining chemical to produce high air contents. Trial mixtures are recommended to assure that proper levels of air can be entrained into a given concrete mixture before it is used on the job.

Corrosion-Inhibiting Admixtures—No Standard. Typically employed to chemically protect steel reinforcement against corrosion-causing agents like chloride, some of these products can also accelerate the hydration process. In this study, corrosion inhibitors were used for their effect on depressing the freezing point of the concrete.

Both set-neutral and set-accelerating corrosion inhibitors based on calcium nitrite were evaluated.

Shrinkage-Reducing Admixtures—No Standard. Aptly named, these admixtures reduce shrinkage in concrete as it hardens, helping to lower the internal stresses that can lead to cracking. In cold climates, fewer cracks make the concrete less susceptible to freeze—thaw damage and prevent ingress of detrimental chlorides. However, shrinkage

reducers were evaluated primarily for their effect on freezing point depression. Shrinkage reducers also provide additional fluid to the concrete, without adding water. This fluid, which does not react with cement, lessens slump loss by keeping the concrete workable longer.

Some shrinkage-reducing admixtures in this study tended to affect entrained air. Some led to low air contents, while others tended to entrain air into the concrete.

#### Specific Admixtures

Concrete admixtures from two U.S. manufacturers—W.R. Grace & Co. and Master Builders, Inc.—were used in this study to develop an antifreeze capability. Table 2 provides a summary of the initial products selected from a much more extensive list of each company's product line. Note that some admixtures are included in two standard categories, reflecting multiple effects or enhanced performance with dosage.

Table 2. Admixtures initially considered for this study.

Standard	Function	W.R. Grace & Co.	Master Builders, Inc.		
ASTM C 494 Type A	Water-reducing	WRDA <sup>®</sup> 82 WRDA <sup>®</sup> with Hycol <sup>®</sup> Daracem <sup>®</sup> 19 Daracem <sup>®</sup> 55 Daracem <sup>®</sup> 65 Mira <sup>™</sup> 70	Polyheed® 997 Pozzolith® 122-N** Pozzolith® 322-N Rheobuild® 1000 Rheobuild® 3000FC Glenium® 3000 NS		
ASTM C 494 Type B	Retarding	Daratard® 17	Delvo <sup>®</sup> Stabilizer* Pozzolith <sup>®</sup> 100-XR		
ASTM C 494 Type C	Accelerating	DCI <sup>®</sup> PolarSet <sup>®</sup>	Pozzolith® 122-HE** Pozzolith® NC 534 Pozzutec® 20 Pozzutec® 20+ Rheocrete® CNI		
ASTM C 494 Type D	Water-reducing and retarding	Daratard <sup>®</sup> 17 Recover <sup>®</sup> *	Delvo <sup>®</sup> Stabilizer* Pozzolith ® 100-XR		
ASTM C 494 Type E	STM C 494 Type E Water reducing and accelerating		Pozzolith <sup>®</sup> 122-HE** Pozzutec <sup>®</sup> 20 Pozzutec <sup>®</sup> 20+		
ASTM C 494 Type F	High-range water- reducing	Daracem <sup>®</sup> 19 Daracem <sup>®</sup> 100 Mira <sup>™</sup> 70 Adva <sup>®</sup> Flow Adva <sup>®</sup> 100	Polyheed <sup>®</sup> 997 Rheobuild <sup>®</sup> 1000 Rheobuild <sup>®</sup> 3000FC Glenium <sup>®</sup> 3000 NS		
ASTM C 494 Type G	High-range water- reducing and retarding	Daracem® 100			
ASTM C 260	Air-entraining	Daravair® 1000 Darex® II AEA	MB AE <sup>®</sup> 90 MB-VR <sup>®</sup> Standard Micro-Air <sup>®</sup>		
None	Corrosion-inhibiting	DCI <sup>®</sup> DCI <sup>®</sup> S	Rheocrete ® CNI		
None	Shrinkage-reducing	Eclipse <sup>®</sup> Eclipse <sup>®</sup> Plus	Tetraguard AS20		

<sup>\*</sup> Hydration control admixture

<sup>\*\*</sup> Contains chloride

Admixtures were chosen based on known performance or physical properties. For each individual admixture, we stayed within the manufacturer's published dosage range. Among the group selected for further study from Table 2, we avoided admixtures containing chlorides, which might lead to corrosion of steel reinforcement. Most of the admixtures contain significant amounts of water that need to be accounted for when designing concrete mixtures.

#### Cement

Throughout the laboratory testing, the same brand of cement was used for consistency. Manufactured by Lafarge North America, St. Constant, Quebec, it met ASTM C 150 (2002a) standards for both Type I and Type II portland cement. A more detailed accounting of its chemical and physical properties is given in Appendix B.

Over the 3-year duration of lab testing, this cement was obtained from a local readymix plant as needed. Potentially, many different lots of cements were included in our overall testing program, but care was taken to ensure that only a single lot was used in any individual trial and each test always included the control mixture as a standard reference point. One exception to this rule was the workability testing (slump), which occurred over an extended period of time and involved many trials. It was, therefore, necessary to use cement from several lots for the slump tests. Nonetheless, the resulting workability profiles obtained provided good indicators of what to expect of the general behavior of each concrete mixture: The results were comparable from test to test.

#### Aggregates and Water

The coarse and fine aggregates used throughout the laboratory test program were obtained from a source local to CRREL in Lebanon, New Hampshire. The coarse aggregate was a 19-mm crushed ledge stone meeting the 67 gradation of ASTM C 33 (2002b). The fine aggregate was a natural sand meeting ASTM C 33 (2002b). Further details on the physical properties of these aggregates are presented in Appendix A.

Tap water from CRREL was used as the mixture water in all test mixtures. The water temperature was allowed to equilibrate with that of the mixing laboratory (approx. 25°C) before being used, either by waiting overnight or by blending hot and cold water.

#### **Concrete Mixture Designs and Formulations**

To find combinations of admixtures meeting our performance criteria, we initially focused on three fundamental issues: workability, plastic air content, and initial freezing point. The amount and type of chemicals used to depress the freezing point of the concrete and accelerate hydration led to issues with both workability and air entrainment. This was not unexpected, given that other high performance concretes in widespread use

today exhibit similar behavior, such as those used in fast-track pavement applications (Zia et al. 1993).

Numerous admixture combinations were evaluated before the field could be narrowed down to a few that met the three criteria simultaneously. More than 50 trial batches representing a total of  $2\frac{1}{2}$  m<sup>3</sup> were mixed over a 2-year period in the laboratory.

#### The Control Mixture

The concrete mixture proportions shown in Table 3 represent the control concrete used throughout the 3 years of laboratory testing. For the antifreeze formulations, the cement and aggregate weights shown below remained unchanged, but the w/c ratio was modified as needed based on the amount of water contained in the admixtures added into the concrete and their effects on workability.

Table 3. Laboratory control concrete mixture design (per cubic meter).

	Mix proportions			
Ingredient	Product description		Amount	
Cement	Lafarge Type I-II	392 kg	(7 sacks)	
Coarse aggregate (ssd)*	ASTM C 33 #67	1079 kg		
Fine aggregate (ssd)	ASTM C 33, fine aggregate	801 kg		
Water	Tap water	171 kg	(171 L) (25 mL /100 kg)	
Air-entraining admixture	Darex <sup>®</sup> II AEA – W.R. Grace	97 mL		
Water-reducer	TM			
	Design Specifications			
Property		Value	Tolerance	
w/c ratio		0.436		
Target slump upon discha	rge	100 mm	±25 mm	
Target entrained air conte	nt (in the plastic state)	6.00%	±1.5%	

<sup>\*</sup> Saturated, Surface Dry. The condition in which the aggregate has been soaked in water and has absorbed water into its pore spaces. The excess, free surface moisture has been removed so that the particles are still saturated, but the surface of the particle is essentially dry

The control mixture represents a typical winter design according to general design considerations established by ACI Recommended Practice 211.1 (1991). Its target slump is 100 mm, as requested by some state DOTs, and it contains 19-mm-maximum size coarse aggregate, which is considered normal size for many concreting applications. According to the guidance from ACI, concrete having this amount of slump and size of aggregate requires a little more than 180 kg/m³ of water. Further, for concrete exposed to freezing and thawing, which most highway structures are, the w/c ratio should be limited to no more than 0.50, preferably 0.45, and its air content should be between 3.5 and 6%

(we chose 6%). To satisfy these constraints, the cement factor of the concrete must be at least  $363 \text{ kg/m}^3$  (we used  $392 \text{ kg/m}^3$ ).

#### Eight Antifreeze Suites (Admixture Combinations)

The initial screening tests, *Laboratory I*, yielded four admixture combinations from each of the two manufacturers. Subsequent verification tests, *Laboratory II*, confirmed that the selected admixture combinations performed properly at low temperatures. Table 4 identifies the resulting eight antifreeze suites. This table shall serve as the key to the test data presented in following sections. Note that brackets are used to indicate that a particular admixture would be added at the jobsite, while no brackets indicates that an admixture could be added into the concrete mixture at the ready-mix plant.

Table 4. CRREL antifreeze admixture combinations (suites).

Deadyst	Admixture dosage ([Brackets] indicate additional dosage or condition at jobsite)						
Product		W.R. Grace & Co. suites (WRG)					
	1	li .	III	IV	Control		
Mira <sup>™</sup> 70 (mL/100 kg)		780	585	390	130	130	
		405	00	65	325	_	
Adva® Flow (mL/100 kg)		195	98		[65]		
DCI® (L/m³)		30	_	30	_	_	
DCI®S (L/m³)			30	_	30	_	
	-		505001	100501	3260		
PolarSet® (mL/100 kg)		[6520] [65	[6520]	[6250]	[2610]		
Daratard® 17 (mL/100 kg)	)		_	<b>-</b> .	260	_	
Eclipse® Plus (% cement	wt)	_	_	1%	_	_	
		60	30		60	25	
Darex <sup>®</sup> II AEA (mL/100 kg)		[30]	[45]	20	[30]	7 25	
w/c ratio (w/o admix)	1	0.317	0.329	0.321	0.303	0.435	
2		0.390	0.400	0.390	0.400	0.436	
w/c ratio (incl. admix)	3	[0.442]	[0.452]	[0.442]	[0.421]	N/A	

		Master Builders, Inc. suites (MB)						
		I	11	III	IV	Control		
Polyheed® 997 (mL / 100 kg)		780	780	390	780			
Glenium® 3000 NS (mL/100 kg)		195	195	65	_			
Rheocrete® CNI (L / m³)		30	30	30	30	]		
Pozzutec <sup>®</sup> 20 (mL / 100 kg)		5870	[5870]	[5870]		OF		
Pozzutec® 20+ (mL / 100 kg)		_	_	_	5870	E OF		
Pozzolith®100-XR(mL/100kg)		_	65	_	_	DUPLICATE CONTROL A		
Tetraguard AS20 (% cement wt)		_	_	1%	_	7 2 5		
MB-VR <sup>®</sup> Standard (mL/100 kg)		40	20	60	20			
		[20]	[20]	[60]	20	GRACE		
w/c ratio (w/o admix)	1	0.316	0.316	0.320	0.271	] %		
/a ratio (in al. a dreix)	2	0.430	0.390	0.390	0.390	W.R.		
w/c ratio (incl. admix)	3	[0.430]	[0.431]	[0.431]	N/A	≥		

Table 4 (cont'd). CRREL antifreeze admixture combinations (suites).

Up to three w/c ratios are provided for each of the eight antifreeze concrete mixtures. Ratio 1 indicates the w/c ratio of the concrete minus the admixtures. This ratio is important to the ready-mix plant as it can be used to calculate the amount of water that the plant operator should add into the initial concrete mixture (minus the free moisture in the sand and coarse aggregate, as usual). Ratios 2 and 3 represent the concrete mixture as it contains some or all of the admixtures. These ratios are important to the engineer as they can be used to calculate the percent solids of the admixtures present in the total water in the concrete mixture (discussed later). Ratio 2 represents the situation where some or all of the admixtures are added into the concrete mixture at the ready-mix plant. Ratio 3, shown in brackets, represents the condition after the final dose of admixture, also shown in brackets, is added into the concrete mixture at the jobsite.

Across the two product lines, some combinations with similar design philosophies were explored. Suite I using W.R. Grace & Co. products (WRG I) and suites I and IV, Master Builders, Inc., (MB I and MB IV) were attempts to create the "fastest" setting mixtures possible from the products available. These suites were based on full doses of corrosion inhibitors and accelerators (Type C or E, or both).

Suites WRG II and MB II were developed to "slow" down the setting of the above suites in an attempt to keep them somewhat workable during transit. WRG II employed a set-neutral corrosion inhibitor, while MB II used a small dose of set-retarding admixture (Type B) to achieve this effect.

Both the WRG III and MB III suites utilized shrinkage reducing admixtures to improve workability of the mixtures. Shrinkage reducers are typically dosed in large enough quantities that they can also contribute to depressing the freezing point of a mixture. They allowed fewer water-reducing admixtures to be used in these mixtures.

The WRG IV suite was an attempt to use a moderate dose of set-retarding admixture to improve workability with both set-neutral corrosion inhibitor and some accelerator aboard during the transport period.

Plasticizers were used throughout the group of eight suites to keep w/c ratios low, and thus maintain a high concentration of freezing-point depressing chemicals.

#### Selecting Effective Combinations (Laboratory I)

This phase tested one batch of concrete for each of the eight prospective admixture combinations under investigation. From each batch, three screening tests were done simultaneously to determine if the concrete remained workable over time, could be entrained with air, and had a satisfactory freezing point.

The admixture dosing sequence in the laboratory was developed to simulate field conditions. For example, admixtures are typically dosed into the concrete both at the ready-mix plant and at the jobsite. Also, most ready-mix plants are situated to service customers up to 1 hour away from the plant. Thus, the laboratory testing considered numerous dosing options, including adding a portion of the admixture into the concrete during initial mixing (to simulate plant addition), waiting for a period of time (to simulate transit time), and then adding the rest of the admixture at a later time (to simulate jobsite addition).

#### Workability (Slump) Over Time

Approaches for "Good" Workability. An important goal for any ready-mix concrete producer is to supply their product to the customer with sufficient workability when discharged at the jobsite. This project used slump, ASTM C 143/C 143M (2000a), to measure workability.

Figure 5 reveals the slump loss over time in a control mixture when it travels down the road to a jobsite without extra water being added into the truck. This provides a baseline of how normal concrete performs over time. It was obtained by measuring slumps from a 0.042 m³ batch of control concrete held in a rotary-drum mixer using the procedure described in the next section. As can be seen, the control concrete started out with the required 100 mm slump but after 45 minutes it dropped to around 50 mm. After 90 minutes the control concrete's slump had dropped to approximately 35 mm. It was unacceptably stiff. To improve its workability, this concrete would have been tempered with water to increase its slump and bring it back to a workable condition.

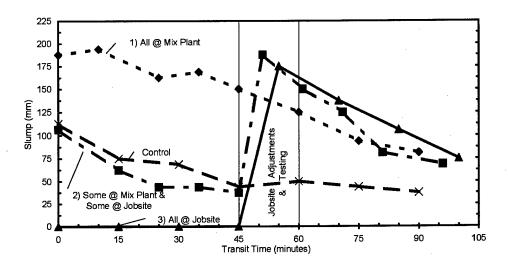


Figure 5. Three methods to achieve workable concrete at the jobsite.

For antifreeze concrete, adding water is not an option, as excess water changes the freezing point of the concrete, making it more susceptible to freezing. The antifreeze concrete, because it contains high doses of accelerating admixtures, is prone to more rapid slump loss than is control concrete. Thus, another approach to achieving workability was needed. We realized that the ability to properly place and finish concrete depends on the slump when it is discharged from the truck, not while it is in transit to the jobsite. Therefore, the slump of the concrete as it was discharged from the truck became the critical issue.

Three approaches for dosing the admixtures into the concrete were devised to assure that the concrete would be easy to work with at the jobsite. Each approach had to assure that the concrete would not stiffen in transit beyond the point that it couldn't be made workable again. That did not mean that the concrete could not lose its entire slump while in transit, it just could not take a permanent set. The pros and cons of the three approaches are shown in Table 5 and briefly discussed next. It should be noted that these three approaches evolved through a series of test batches made up of numerous combinations of the admixtures shown in Table 4. It was envisioned at the start of the project that this type of testing would be necessary because many of the admixtures chosen for study contained hydration accelerating chemicals and rapid slump loss was considered a possibility. The three approaches discussed below were tested numerous times in the laboratory while, only a few where actually used in the field as noted later.

Table 5. Tradeoffs among three admixture dosing approaches.

•	Approach							
Issue	1—Plant	2—Plant/jobsite	3—Jobsite					
Transit time	Long periods not feasible	Somewhat longer than approach 1	Longest times possible					
Tailoring initial slump for different haul times	Recommended for achieving target slump at jobsite	May be necessary with some mixtures	Minimal					
Slump loss and stiff- ening in transit	Greatest	Moderate	Minimal—same as "normal" concrete					
Effects of ambient or mixture temperatures	Greatest variability and slump loss if high	Moderate variability and slump loss if high	Minimal, however, must be careful con- crete without admix- tures doesn't freeze in transit					
Transit delay	Could lead to signifi- cant stiffening and no slump	Could lead to prob- lems with stiffening and low slump	Minimal problems— no admixtures to cause slump loss					
Onsite dosing equipment and personnel	None needed	Some required	Most needed —may require separate set- ups for different ad- mixtures					
Onsite corrections or adjustments	Not prepared for any deficiencies	Limited to admix- tures on hand	Total control over adjusting mixture properties					
Waiting time for job- site additions	None needed before placement	Fewer additions require less time	Demands the most attention and time					
Concrete placement	Rapidly stiffening mixture may require quick placement and finishing	High slump after job- site additions may require waiting be- fore placement be- gins	Greatest possible working times onsite					
Variability in deliv- ered product			Least potential vari- ability, especially when pre-tested and dosed properly					

Approach 1, dosing all the admixtures into the concrete at the ready-mix plant, has the advantage that once the truck leaves the plant, no further effort is needed to prepare the concrete. However, because slump loss will be rapid, it is necessary that the concrete leave the plant with a high enough initial slump so that when it arrives at the jobsite it will still be workable. This suggests that haul time be as short as possible to avoid prob-

lems caused by unexpected delays along the way. Curve 1 in Figure 5 illustrates this point by showing the lab slump test data from a mixture very similar to MB I, but starting with a higher slump.

Approach 2, dosing some of the admixtures into the concrete at the ready-mix plant and the rest at the jobsite, has the advantage that slump loss is less of a concern. The admixtures that have little effect on slump loss can be dosed first, followed by those that do. This requires that admixtures be transported to the jobsite and pumped into the truck once the truck arrives. Though requiring some extra effort, this method provides more assurance that the concrete will be workable and unforeseen delays will have less effect. Curve 2 in Figure 5 illustrates this approach, showing the performance of WRG I in a lab slump test.

Approach 3, dosing all the admixtures into the concrete at the jobsite, has the advantage that slump loss is not an issue. Once the admixtures are dosed into the concrete, the concrete is ready for placement. However, because the admixtures contain water, the concrete must begin with a very low w/c ratio at the ready-mix plant. As with approach 2, the admixtures must be transported to the jobsite and dispensed there. This requires more effort at the jobsite but normal construction delays are not a problem as the admixtures do not have to be dosed until the concrete crew is ready for it. Curve 3 in Figure 5 represents the performance of the MB IV mixture in the lab slump test. The start time has been "shifted" ahead 45 minutes in time to simulate the performance of the suite once the admixtures are dosed at the jobsite.

*Procedure*. To test for workability over time, we mixed small batches (0.042 m<sup>3</sup>) of antifreeze concrete in a revolving drum mixer (Fig. 6) and periodically measured it for slump. The testing was done at room temperature (25°C) to simulate what might happen in larger volume field mixtures that could warm up during transit.

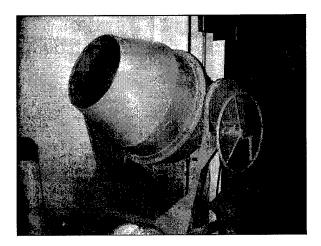


Figure 6. Revolving drum mixer in the laboratory.

After the mixer was pre-buttered with mortar, the drum was charged with aggregate to begin the mixing process. Each admixture was dosed separately into the concrete, to avoid direct contact with other admixtures, as recommended by manufacturers. Appendix B details how batching, mixing, and fresh concrete testing were conducted.

The first slump was measured immediately following initial mixing by removing a small quantity of concrete from the mixer. The material used in the slump tests was returned to the mixer after each test. The concrete was mixed for 45 seconds every 5 minutes to approximate a slow agitation of about 4 rpm during delivery. Slump measurements were repeated at 15, 25, 35, and 45 minutes following the first measurement. (Alternately, for test mixtures that did not require a jobsite dose of admixture [Table 4], slump measurements were taken at 15-minute intervals.)

After the concrete from the 45-minute slump test was returned to the mixer, jobsite admixtures were dosed into the concrete and mixed for 2 minutes to ensure thorough incorporation into the mixture. Slump was measured immediately following mixing and at 15, 30, 45, and 60 minutes thereafter. Remixing every 5 minutes continued during this period until the test was complete.

Results and Discussion. Table 6 presents the slump readings for each of the eight antifreeze concrete mixtures as well as that for the control.

Time from end of initial mixing (minutes)															
	esigns	0	15	25	30	35	45	50	60	70	75	80	90	95	110
:	WRG I	110	65	45		45	40	190	150	125	_	85	_	70	0
	WRG II	100	70	55		40	40	180	135	110	_	75	_	55	40
	WRG III	65	65	65	_	55	50	215	180	170		140	_	85	25
Œ.	WRG IV	240	205	180		140	95	215	205	180	_	150	_	115	65
Slump (mm)	Control	115	75	_	70	_	45		50		45		40	_	_
Slun	MBI	140	140	_	110	_	75		65		55		45		
	MB II	85	65	65		55	50	165	140	125	_	100	_	95	55
	MB III	30	30	30		25	25	230	205	190	_		_		_
	MB IV	180	140	_	110		75		50	_	40		40		
Transit period Ad					Adjust	ments		Wor	king p	eriod					

Table 6. Slump values over time for test mixtures.

As can be seen, WRG I, WRG II, and MB II best achieved the target slump of 100 mm for the 20- to 30-minute work period following 45 minutes of simulated transit plus

10 minutes of jobsite adjusting time. A rectangular box is used to denote this period of interest in Table 6. It should be noted that, in some cases, the slumps started out relatively high but ended up reasonably low at the end of the working period. These concrete mixtures employed Approach 2 of dosing some admixtures at the ready-mix plant and the rest at the jobsite (Table 5).

On the other hand, MB I and MB IV did not meet our criteria. Their slump levels dropped below the 75 mm threshold after 45 minutes, ahead of the crucial placement period. However, because these concretes, which employed dosing approach 1 (Table 5), retained much of their initial slump for up to 45 minutes, they could be very useful for jobs that require short haul times. Conversely, as we show later in the field studies, dosing all the admixtures at the jobsite (approach 3, Table 5) for these suites assures a very workable concrete where it is needed the most, at the jobsite.

Both MB III and WRG III contained shrinkage-reducing admixtures, which considerably improved the performance (Table 6) of the concrete in transit. However, once the site doses of admixtures were introduced, these mixtures gave very high slumps that took too long to fall into our target range. Because these mixtures react so positively to the jobsite additions when it comes to slump, it does not appear the early setting is a concern. Thus, a solution that should be tried is to withhold more water from the initial mixture. Also, because they contained the shrinkage reducing chemicals, with these suites, it was difficult, and in some cases impossible, to achieve concrete with good amounts of entrained air. This shortcoming is addressed further in the following section.

WRG mix IV was an attempt to use a set-retarding admixture to better control slump loss in transit. With a majority of the active, set-accelerating admixtures added at the beginning, we hoped a retarder might counteract the tendency for the mix to stiffen in transit. Unfortunately, even though the mix started out at a high slump, it still lost a considerable amount of slump over the transit period. Plus, the final jobsite admixture doses yielded a high slump concrete that required a long waiting period before it fell into the target slump range.

These tests provided us with the confidence that we could formulate large batches of concrete in field conditions without serious concern over losing a batch. Though not all of the tests proved successful in the laboratory, they provided sufficient insight for us to experiment further in the field.

#### Entrained Air in the Plastic State

Current guidance requires the air entrainment of all concretes exposed to freezing and thawing environments. We used an air-entraining admixture to produce a system of non-interconnected bubbles within the paste matrix of the concrete. The target air content was  $6 \pm 1.5\%$ .

Air contents were measured twice on each of the concrete mixtures in this study using the volumetric method, ASTM C 173 (1994), because of the smaller sample size it requires. The first air content sample was obtained within 5 minutes of having fully mixed concrete. The second test was done 50 minutes following the first measurement to determine if the air content changed over time. For the concrete mixtures that were dosed with jobsite admixtures, the second air content was taken immediately following that addition. For those concrete mixtures, where all admixtures were dosed at the ready-mix plant, the second air content was tested at the 55-minute mark. The results are presented in Table 7.

Table 7. Entrained air measurements over time.

	Air content (%)					
Admixture suite	5 min. after mixing	55 min. after mixing				
WRG I	4	7.75				
WRG II	4	~ 9.5				
WRG III	3	~ 11.75				
WRG IV	5.25	8				
Control	7	5.25				
MB I	4.25	4.25				
MB II	4.5	4				
MB III	2	2.75				
MB IV	~ 11	5.5				

Note that in many cases, the air contents of the antifreeze concretes were well over the 6% target value. Though high air contents do not necessarily imply good air void systems, high air contents were welcome news, as they meant that the admixtures did not prevent air from being entrained into the concrete. In fact, they may have enhanced it. In some cases, the air contents increased with continued intermittent mixing, while in others it decreased. As discussed in the section on freeze—thaw durability, the tendency to lose air, particularly when the concrete is continually worked during sampling, has implications for laboratory studies though probably not for field work. At this point, we were only checking to see that these mixtures would accept entrained air. Adjustments were made later to get better overall air content values during subsequent strength, freeze—thaw, and field trials.

#### Initial Freezing Point

The freezing point depression test is used to verify that fresh antifreeze concrete is protected from freezing, in this case down to an internal concrete temperature of at least -5°C.

Figure 7 illustrates typical cooling curves for control concrete and antifreeze concrete. In the laboratory, cylinders of fresh concrete are placed into a –20°C coldroom to cool. The curves illustrate how the temperature changes as heat is extracted from the concretes. Both curves show that concrete steadily cools from room temperature to the point where ice suddenly forms. The freezing point on each curve is identified as the location where the slope of the cooling curve begins to flatten. At this point, water in the mixture is slightly supercooled, meaning that this temperature is lower than that required for ice to melt (Alexiades and Solomon 1993). When ice crystals form, there is a slight increase in the temperature (a matter of tenths of °C) caused by the release of latent heat of fusion. For the control concrete, ice continues to grow at a constant temperature until all water has turned to ice. For the antifreeze concrete, the water does not freeze at one temperature. This is because the solid that freezes out from solution is pure ice. As ice develops, the concentration of admixtures in the remaining water increases. Thus, progressively lower temperatures are required to freeze out more ice.

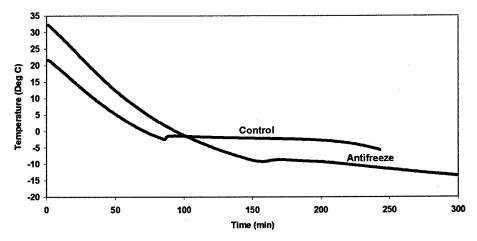


Figure 7. Typical cooling curves for control and antifreeze concretes.

*Procedure.* Freezing points were determined by embedding thermocouples into cylinders of fresh concrete placed into a  $-20^{\circ}$ C room. Three cylindrical samples were cast in 76-  $\times$ 152-mm plastic molds from each concrete batch and fitted with a thermocouple at the center of mass. The cylinders were then capped and placed into the coldroom where temperatures were recorded every 60 seconds with a datalogger. When the initial sample

temperature was 20°C or lower, the sample cooled off at a rate of approximately -0.5°C per minute, which allowed the freezing point to be determined within 60 to 70 minutes.

Three test cylinders were chosen. When all three of the curves were within 0.5°C of each other, the freezing point temperature was determined as the average of the three measurements. If one freezing point measurement deviated more than this amount or could not be seen clearly in the data, it was dropped and the average of the two similar measurements was reported.

Careful review of the data was important to assure that the freezing point temperature was not misinterpreted (see Appendix C for a full description of the freezing point procedure).

Calculating Concentrations. A relationship exists between the concentration of admixture in the mixing water and the freezing point of the fresh concrete. This relationship comes from the proportion of the total solids to the total free water present in the concrete. The lower the concentration, as might be caused by a higher than desired water content in the concrete, the higher the freezing point becomes (Fig. 8). Varying the total percent solids and measuring the resulting freezing point generates a range of freezing point readings. Plotting the values generated under laboratory conditions provides a useful way to check on freezing points in the field. Corrective action may be taken to increase the total percent solids content to ensure proper freeze protection should the mixture freezing point, as measured in the field, be higher than the design specification.

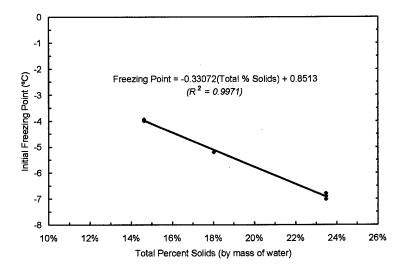


Figure 8. An example of the linear relationship between total percent solids by mass of free water and the initial freezing point for MB I antifreeze concrete.

All of the water in the concrete mixture is accounted for by using a percent solids calculation. The weight of solids and water contributed by each admixture is calculated (eq 1 and 2). The total water from all admixtures is then added to the mixing water (based on saturated, surface dry [ssd] aggregate) to obtain the total free water weight (eq 3). The total mass of solids from all admixtures is finally divided by the total free water weight to give the total percent solids for the antifreeze concrete.

$$Solids_{i} = Admixture_{i} \times Percentsolids_{i}$$
 (1)

where:

 $Solids_i$  = mass of the solids for an individual admixture (kg)

 $Admixture_i$  = total mass of a single admixture (kg)

 $Percentsolids_i = percent of solid material (by weight) in a given admixture.$ 

$$Water_{i} = Admixture_{i} \times (1 - Percentsolids_{i})$$
 (2)

where Water; is the mass of water for an individual admixture (kg).

$$Total percent solids = \frac{\left(\sum_{i=1}^{n} Solids_{i}\right)}{\left(\sum_{i=1}^{n} Water_{i}\right) + Mixwater}$$
(3)

where:

Total percent solids = total solids content for the antifreeze mixture

Mixwater = mass of water (saturated surface dry) used in the mixture (kg)

n = total number of admixtures in the antifreeze mixture.

Results and Discussion. During the workability tests, concrete mixtures were tested to see if they met our freezing point target of at least -5°C. Samples were taken from the mixer once the initial mixing was completed and tested according to the method introduced in this section on initial freezing-point. A second set of samples was taken immediately following any jobsite admixture additions. The second measurements were complicated because of the need to account for the concrete removed from the batch during prior testing. Nevertheless, the freezing point measured was valuable as a guide and assured that we were close to our goal.

Subsequent freezing point measurements made during strength, set time, and freezethaw beam casting—where no material was removed prior to all admixture doses being made—confirmed what we found originally in the slump trials. All of the concrete mixtures met or surpassed the -5°C goal. A summary of the results obtained while casting strength samples is presented in the central columns of Table 8, representing the suites' performance in depressing the freezing point of the concrete.

As a guide to the relationship between the concentration of the admixtures in the concrete and their freezing point depression, additional tests of mortars at w/c ratios 0.1 above and below the desired level were performed. These results, to the right and left of the central columns in Table 8, show the effect that variations in water content, or dosages of the admixture suites, can have on the percent of total solids in the water, and thus the initial freezing point of the concrete. This relationship between percent total solids and the freezing point can be plotted linearly and used to predict freezing points of different dosages. Alternately, if the freezing point is measured, the percent of solids in the mixture can be estimated and used to back-calculate what the actual water content of the mixture is. We found this to be an invaluable tool in our field testing to determine the w/c ratio of the mixture when the control over moisture in the aggregates was questionable. Figure 8 shows an example of plotting the relationship for MB I. Users are urged to determine this relationship using the values for the particular concrete mixture they use. If other proportions of the admixture dosages are used, then testing must be done to determine the new association.

Table 8. Effect of w/c ratio on laboratory freezing point measurements and percent total solids contents for antifreeze concrete. Note that when the design w/c ratio is met, all antifreeze suites meet or surpass the -5°C goal.

Admixture suite	1	wer than get	Target w/c ratio		w/c 0.1 higher than target	
	Initial freezing point (°C)	Total % solids** (by wt of water)	Initial freezing point (°C)	Total % solids (by wt of water)	Initial freezing point (°C)	Total % solids (by wt of water)
WRG I	-6.3	20.71	-5.5	16.03	-4.5	13.07
WRG II	-6.1	19.76	-5.2	15.39	-4.4	12.60
WRG III	-6.7	23.21	-5.8	17.96	-4.5	14.65
WRG IV	-6.2	20.96	<b>-</b> 5.7	15.99	<del>-4</del> .1	12.92
Control	_		-1.0*	0.08	_	_
MB I	-6.9	23.48	<i>–</i> 5.2	18.04	<del>-4</del> .0	14.62
MB II	-6.9	23.52	-5.2	18.07	-3.9	14.67
MB III	-7.2	25.60	-5.3	19.66	<del>-4</del> .0	15.96
MB IV	-7.4	23.60	<b>-</b> 5.5	17.55	-4.5	13.97

<sup>\*</sup> Korhonen (2002) shows that w/c ratio has little effect on freezing points of control concrete.

<sup>\*\*</sup> Solids % calculated using eq 3.

# Confirming Low Temperature Performance (Laboratory II)

With the concrete mixtures that passed (Table 4) the three initial screening tests (slump, air content, and freezing point), further testing was done to evaluate how well they performed against the remaining criteria. This round of testing included those introduced in Figure 4:

- Compressive strength development.
- Freeze-thaw durability.
- Time of setting.
- · Critical maturity.

These tests served to demonstrate that the concrete mixture proportions developed in the *Laboratory I* phase would perform as intended at low temperatures.

# Compressive Strength Development at 25°C, 5°C, and -4°Ct

Procedure. The purpose of this test was to determine how quickly the concrete would gain strength over a wide range of temperatures. Concrete was mixed at room temperature according to the procedure outlined in Appendix B, through the 8-minute mark. For concrete mixtures that required jobsite additions of admixtures, waiting periods before and after the dosing allowed the concrete to achieve a reasonable slump and be cohesive at the time of casting. As with slump testing, the mixture was agitated for 45 seconds every 5 minutes during these rest periods. The concrete was cast at room temperature (25°C) into 76- × 152-mm plastic cylinder molds in accordance with ASTM C 192/C 192M (2002c) and external vibration was used to consolidate them. One exception was that the cylinders were cast in two layers, instead of the standard three, to ensure prompt sample preparation with any accelerated mixtures. All strength samples for each suite were cast from the same batch of concrete and sealed with caps to prevent evaporation. The plastic air content was measured for each set of cylinders cast using the volumetric method (ASTM C 173 [1994]).

All samples were placed on wire shelves in the curing rooms 80 to 115 minutes after the mixing water was added. For each suite, companion dummy cylinders containing thermocouples at their centers of mass were placed in each curing room to monitor temperature history (Fig. 9). As seen in Figure 9a, the samples in the 5 and the -4°C rooms rapidly cooled off over the first 3 hours before slowly cooling off to their final temperature over the next several hours. These cooling times could have been sped up had water

<sup>&</sup>lt;sup>‡</sup> Although the freezing point of the antifreeze concrete in this study was at least  $-5^{\circ}$ C, the coldroom temperature was set at approximately  $-4.5^{\circ}$ C to account for the normal  $\pm 0.5^{\circ}$ C thermal

baths been used, but these results sufficiently tested our concretes. For example, the -4°C samples reached -3°C within 6 hours after being placed into the coldroom. This is well below the freezing point of control concrete (Table 8) and well before all but one of the antifreeze concretes initially set (Table 14). Getting the cylinders into the coldroom, a full hour before the fastest initial set time recorded ensured that the fresh concrete had to resist freezing and that the majority of the resulting strength gain took place at the specified curing temperature. Figure 9b shows that some concretes were maintained at their two lowest curing temperatures for 28 days before being returned to room temperature.

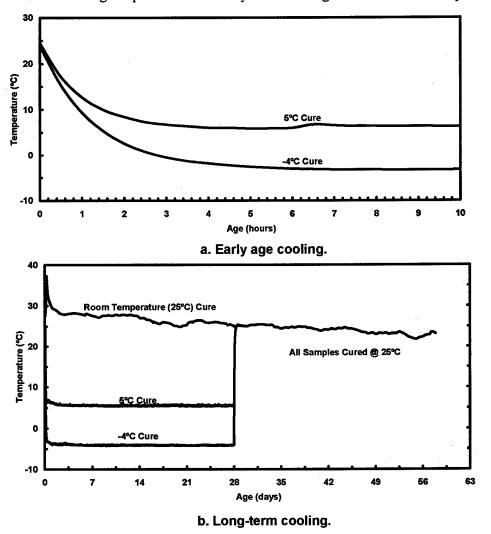


Figure 9. Typical temperature history for strength cylinders.

cycling of the room. This assured that no ice formed in any sample. This setting caused the test samples to cure at slightly less than  $-4^{\circ}$ C over the curing period.

Table 9. Compressive strength of antifreeze concretes (in percent relative to 25°C 28-day control).

	Cured at 25°C								
	1 day	3 days	7 days	14 days	28 days	56 days			
WRGI	52.5	_	117.4	129.0	137.5	_			
WRG II	46.1	_	93.4	105.0	110.3	_			
WRG III	55.3	_	97.3	112.9	110.7				
WRG IV	73.4	_	119.8	133.7	145.9				
Control	47.1	-	87.4	96.2	100.0	105.0			
MB I	75.5	_	127.7	135.7	153.4	_			
MB II	80.4	_	130.9	142.2	155.8	_			
MB III	72.1	_	131.1	138.8	152.9				
MB IV	83.7	_	135.7	145.6	158.5	—			
	(	Cured at 5	°C for init	ial 28 days	3				
WRGI	19.2		80.5	104.6	121.9	165.4			
WRG II	11.1		53.0	82.0	104.0	135.9			
WRG III	21.6		71.9	89.0	108.1	132.0			
WRG IV	9.7	<del>-</del>	98.7	125.3	155.1	171.1			
Control <sup>†</sup>	2.0	15.0	30.0	55.0	80.0	100.0			
MB I	21.7	_	111.5	137.6	157.9	176.1			
MB II	13.3	_	108.6	133.8	159.1	182.1			
MB III	18.9	_	104.0	128.0	142.5	173.2			
MB IV	31.8		104.2	131.4	149.7	172.5			
	C	ured at	4ºC for ini	tial 28 day	s				
WRGI	1.6		55.8	69.7	98.9	170.3			
WRGII	0.3		23.6	41.8	57.4	134.9			
WRG III	2.5		51.9	67.2	90.4	136.7			
WRG IV	0.5	_	38.3	81.5	120.6	178.8			
MBI		32.9	76.9	105.2	125.4	181.9			
MB II	_	20.7	68.6	98.4	123.7	174.6			
MB III		21.9	65.6	89.7	118.3	176.2			
MB IV	_	24.2	71.2	100.9	119.0	176.3			

<sup>&</sup>lt;sup>†</sup> After ACI 306 (1988).

Samples remained in the capped, plastic molds until tested. At various ages (Table 9), sets of three cylinders were removed from the curing rooms, demolded, and allowed to warm to 5°C at their center of mass, when necessary. This warming, which took about an hour, ensured that no specimen contained ice during testing, which could incorrectly lead to higher strengths, and that no unnecessary hydration could take place before strength

testing. Unconfined compressive strength testing was then carried out according to ASTM C 39 (2001b) using unbonded neoprene end caps (ASTM C 1231 [2000b]). The cylinders remained in each room until tested or until 28 days. After 28 days all untested cylinders were moved to room temperature (25°C) for an additional 28 days of curing (recovery period). This additional curing showed whether the freezing temperatures had caused any permanent strength loss.

Results and Discussion. The results of the compressive strength testing of the eight antifreeze mixtures and the controls are presented in Table 9. All values are given in percent, relative to the 28-day strength of a companion control mixture that was made, cured, and tested at 25°C. The W.R. Grace mixtures were run in one trial and compared with a companion control mixture, while the Master Builders mixtures were run in a separate trial—including its own control. As a baseline for the performance of antifreeze concrete, we chose to use the well-accepted values of strength-gain rate for a typical Type I cement concrete when cured at 40°F (4.4°C) given by ACI 306-R88 Section 6.6.1 (1988). This baseline is clearly shown in Table 9.

When cured at 25°C (room temperature), the antifreeze concretes developed greater strength than the control concrete at all ages. At 28 days, the antifreeze concretes performed significantly better than the control, providing an additional 10 to 60% more strength. It is not clear why this occurred, but it is good news. The importance of the room temperature study is that all antifreeze concretes provided higher ultimate strengths than the control. This strongly suggests that strength development in antifreeze concrete should not be harmed by unexpected periods of warm weather that occur after placement.

When cured at 5°C, the antifreeze concretes developed less strength at 1 day compared to the room temperature control, but significantly more than the 5°C control. At 7 days and beyond, the antifreeze concretes performed as well as the 25°C control concrete. By 28 days all antifreeze concretes had exceeded the 28-day strength of the control concrete and had practically equaled their own strength attained at room temperature. And, by 56 days, the antifreeze concretes produced an extra 30 to 80% strength. That is significant, and, as we will see, that advantage carries over to lower temperatures as well.

When cured at -4°C, the antifreeze concretes essentially gained strength as rapidly as did the control concrete cured at 5°C, satisfying one of the primary goals of this study. The only exception to this was that the concrete made with the WRG II admixture combination gained only about 75% as much strength compared to the 5°C control over the first 28 days. Though it is not clear why this happened, a possible explanation for this shortfall is that the WRG II used fewer accelerating admixtures than the other admixture combinations tested. Nevertheless, the WRG II concrete, like the other antifreeze concretes, ultimately outperformed the control concrete. At 56 days the antifreeze concretes produced the same 35 to 80% extra strength that they did at 5°C. This shows that no damage took place to any of the antifreeze concretes cured under these conditions.

# Freeze-Thaw Durability

Introduction. Freeze-thaw durability tests were conducted as part of the performance tests on the candidate antifreeze admixtures. The objective was to verify that the admixtures did not harm concrete by preventing air from being entrained into concrete. Testing was conducted using ASTM C 666, Procedure B (1997a).

Procedure. The first durability test evaluated concrete made with W.R. Grace products. For each of the four concrete mixtures, two sets of test beams were cast: non-air-entrained and air-entrained. Air content measurements were taken of the fresh concrete in accordance with ASTM C 173 (1994). The non-air-entrained beams were mixed and cast on one day and the air-entrained beams were mixed and cast 6 days later. All beams were cured in room temperature limewater for 28 days. The beams were then rinsed in tap water and set aside in a 23°C, 50% RH room. The non-air-entrained beams remained in the warm room for a total of 11 days, and the air-entrained beams for 5 days. Once all beams were cured, they were soaked in water overnight and subjected to freeze—thaw cycling.

The concrete beams made with Master Builders products were prepared similarly to the W.R. Grace beams.

Results. Figure 10 shows the results from the four non-air-entrained W.R. Grace antifreeze concretes compared to that of the control concrete. As can be seen, for all but the WRG IV concrete, the antifreeze concretes behaved similarly to that of the control concrete, illustrating that the admixtures do not degrade the freeze-thaw durability of concrete. This makes sense because all of the admixtures used in this study are commercial products and have been tested by their manufacturers for their effects on the short- and long-term properties of concrete for them to be approved for general use. (Because the Master Builders admixtures essentially used the same chemicals as those used in the W.R. Grace admixtures, the Master Builders products were not tested on non-airentrained concrete.) Interestingly, the concrete made with the WRG IV admixture combination was significantly more durable than the other concretes. According to ASTM C 666 (1997a), this concrete is considered freeze-thaw durable, as it retained more than 60% of its relative dynamic modulus (RDM) of elasticity after 300 cycles of freezing and thawing, even when non-air-entrained (measurements showed that it contained only 2% air when fresh). One possible explanation for this good showing is that one of the chemicals used in the WRG IV formulation reduces the surface tension of water, which tends to reduce shrinkage forces. This chemical may also reduce the tendency for concrete to rewet once some or all of its pores have dried out. As explained elsewhere in this report, some samples were allowed to partially dry out in a 50% RH room. Normally, this would only dry out the larger pores in the cement paste, so quickly rewetting them is usually not a problem in normal concrete. In hindsight, we cannot say that this concrete, because it contained the shrinkage reducing chemical, was fully saturated during the freeze-thaw testing. This is an interesting question that needs further study. However, these results

suggest that chemical admixtures, at least the ones used in the WRG IV combination, may actually have a positive effect on durability. This is something that also needs further study.

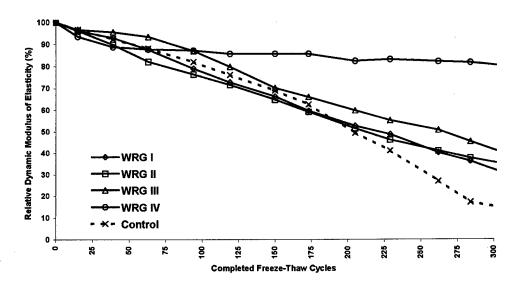


Figure 10. Relative dynamic modulus of elasticity for non-air-entrained mixtures using W.R. Grace admixtures. Each line represents the average values from three replicate beams. (The air contents of the fresh concretes were: WRG I, 1.75%; WRG II, 1.75%; WRG III, 2.0%; WRG IV, 2.0%.)

Figure 11 shows some of the results from the first test of W.R. Grace air-entrained antifreeze concretes. As can be seen, the WRG I, III, and IV concretes were considered durable according to ASTM C 666 guidance because they maintained 60% of their RDM through 300 cycles of freezing and thawing. The WRG I concrete was just able to qualify, while the WRG III and IV concretes were only mildly affected by 350 freeze-thaw cycles. The WRG II concrete, on the other hand, was not durable. Its RDM dropped to 60% at approximately 200 freeze—thaw cycles. Though these results show that antifreeze concrete can be entrained with air and that it can be made durable, we were not sure why the WRG II concrete, which contained 7.5% air soon after it was mixed, was not as durable as the WRG I and IV concretes, which contained only 4.5% and 5.25% air, respectively. Presumably when concrete contains around 5-7% air, appropriately dispersed within the cement paste, it should be fairly resistant to cycles of freezing and thawing in a moist condition. The expectation was that the more air there is, within a reasonable range of course, the more durable the concrete. Two questions immerged: 1) how much air was there and how was it spaced in the hardened concrete? and, 2) do some antifreeze concretes require higher air contents than others to be freeze-thaw durable? To try to answer these questions, a second round of freeze-thaw tests was conducted. This time the

amount of air-entraining agent was increased in each concrete mixture to entrain more air into the concretes. All else remained the same.

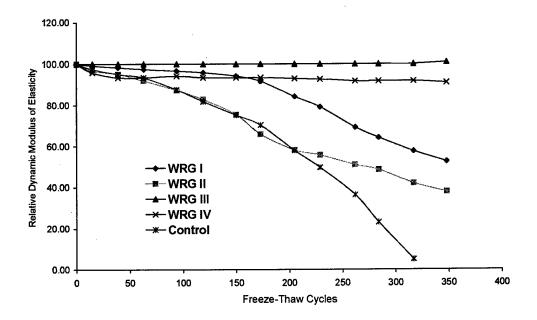


Figure 11. The relative dynamic modulus of elasticity for W.R. Grace air-entrained concretes. Each line represents the results from one beam. (The air contents of fresh concrete are: WRG I, 4.5%; WRG II, 7.5%; WRG III, 8.75%; WRG IV, 4.5%; Control, 1.5%.)

Figure 12 shows the best and worst single-beam results from the second round of freeze—thaw testing of the W.R. Grace concretes. As can be seen, the results varied widely from a beam being considered durable to a replicate beam being not durable. At first this concerned and confused us, particularly because the fresh concretes contained much more air this time around. The interesting thing to note is that the WRG II concrete that was just found barely durable in Figure 11, this time was found essentially unaffected by 300 cycles of freezing and thawing. This finding reinforced our earlier conclusions that antifreeze admixtures do not harm concrete and that concrete made with these admixtures can highly durable. Why there would be such variability between beams from the same batch of concrete was a puzzlement. At this point we had not examined the hardened concrete for air voids.

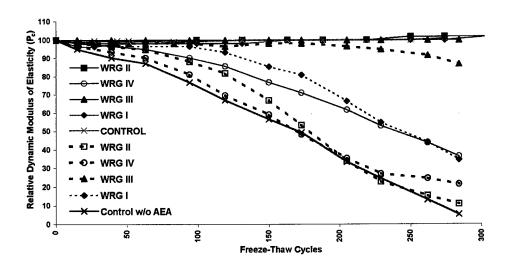


Figure 12. Relative dynamic modulus of elasticity for W.R. Grace air-entrained concretes. The dotted line represent beams with the poorest showings while the solid lines show the best results from replicate beams. (The air contents of two fresh concretes were higher than could be measured: WRG I, >9%; WRG II, >9%; WRG III, 9%; WRG IV, 7%.)

We recalled from our earlier laboratory testing during this study that the air content of fresh antifreeze concrete was more difficult to control than it was for fresh control concrete. The earlier laboratory tests showed that antifreeze concrete could be entrained with air but that it tended to lose air more rapidly over time compared to control concrete—especially if the concrete was worked. In this study, air contents of fresh concrete were measured about 10 minutes after mixing but freeze-thaw beams and strength cylinders, scooped from the same batch of concrete, were continuously made for about an hour thereafter. Thus, it was plausible that freeze-thaw beams made from the same batch of concrete could vary widely in air content from beam to beam. To test this idea we prepared several batches of concrete (WRG I, WRG II, MB IV, and control) in a separate study and measured their air contents immediately after mixing and then again later after simulating the scooping action of making test specimens. Initial air contents of as high as 9% dropped to 3% within about 45 minutes—this was true for both antifreeze and control concrete, where the control concrete seemed to lose air less rapidly. No further testing was done as this demonstrated to us that beams made soon after mixing can turn out to be more durable than replicate beams made some time later. The important message from Figures 11 and 12 is that antifreeze concrete can be made freeze-thaw durable. Any future study should ensure that replicate freeze-thaw beams be made at the same time as opposed to widely different times. More work is needed in this area.

To provide one more check as to whether antifreeze concrete can be entrained with the proper amount and spacing of air bubbles, one replicate beam from each antifreeze mixture from round two testing was sent to W.R. Grace for hardened air-void analysis. These beams were extras that were not subjected to freeze—thaw cycles. Though they could not be used to answer what the air void system was like within the beams that were freeze—thaw tested, the beams sent to W.R. Grace could still provide useful information. W.R. Grace was not informed as to the identity of the beams. Table 10 shows that all of the antifreeze concretes contained entrained air—some more than others—and that all but the WRG IV beam contained a reasonable spacing factor (ASTM considers a beam to be durable if it contains more than 4.5% air and its spacing factor is 0.2 mm or less). Unfortunately, we did not keep track of the exact time that these extra beams were fabricated in relation to the beams that were freeze—thaw tested. Thus, it is impossible to comment on how closely these air contents correlate to air contents of the replicate freeze—thaw beams. This study does, however, suggest that antifreeze concrete is air entrainable. (The CPAR study conducted nearly 10 years ago and discussed later in this report came to the same conclusion.) This study did not include an in-depth assessment of the air void system in the beams that were freeze—thaw tested. Further work is warranted in this area.

Table 10. Air-void analysis of a single beam of hardened concrete not subjected to freeze-thaw testing\*.

Sample	Air (%)	Spacing factor (mm)
WRGI	7.49	0.221
WRGII	6.56	0.221
WRG III	5.07	0.139
WRG IV	5.88	0.312

<sup>\*</sup> Courtesy Neal S. Berke, W.R. Grace.

The results for the concretes made with Master Builders products are shown in Figure 13. As can be seen, the concretes made with MB I and MB II admixture suites slightly exceeded the durability of air-entrained control concrete. After 300 freeze—thaw cycles, both antifreeze concretes retained over 90% relative dynamic modulus of elasticity (RDME) while the control retained slightly less than that value. Concrete made with the MB IV admixture suite on the other hand did not fare as well, dropping to 60% RDME within 150 freeze—thaw cycles. Inadequate air content was not a problem during mixing, but maintaining adequate air throughout the hardening process may have been. As previously discussed, laboratory technique may have caused the entrained air to be lost. The concrete made with the MB III suite also had poor freeze—thaw durability. Its poor performance was about on par with the non-air-entrained control concretes shown in Figures 10, 11 and 12. However, we found that the shrinkage-reducing admixture used as part of this suite tended to greatly detrain air from the mixture, making it very difficult to entrain proper amounts of air into concrete. As the caption to Figure 13 shows, MB III, which contained an air entraining admixture, only contained 1.5% air while fresh, which was

comparable to that found on the non-air-entrained control concretes. These antifreeze beams, later returned to the freeze—thaw chamber for additional exposure, seemed to drop well below an RDME of 60% within only a few more freeze—thaw cycles. Perhaps other air entraining admixtures would produce better results with this antifreeze formulation, but that possibility was not explored in this study. Thus, until additional verification testing is conducted, MB III is the only concrete that we would not recommend for outdoor applications at this time.

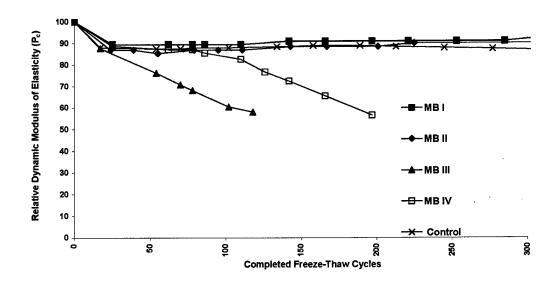


Figure 13. Relative dynamic modulus of elasticity using the Master Builders admixtures. Each line represents the average values from three replicate beams. (The air contents of fresh concrete were: MB I, 6.25%; MB II, 6.25%; MB III, 1.5%; MB IV, 7.5%; control, 8.5%.)

Comparison to a Previous Test. From 1992 through 1995, CRREL partnered with W.R. Grace and Master Builders to explore the efficacy of using chemicals to depress the freezing point of fresh concrete (Korhonen et al. 1997, Korhonen and Brook 1996). This effort was a Congressional initiative for the Corps to work with private industry on research and development that had potential for advancing the art of construction and for being of value to Corps construction activities. The investigations were conducted under the authority of the Corps' Construction Productivity Advancement Research (CPAR) program. The program proved that antifreeze technology worked—producing two prototype antifreeze admixtures—but never advanced beyond that stage because it was felt that an industry acceptance standard should be in place before this new technology could be released to general practice.

Of interest to our study, the CPAR program evaluated the effect of various chemicals, both prototype and off-the-shelf admixtures, on the freeze-thaw durability of concrete. (Because the admixtures were proprietary, the chemicals used in them are not disclosed.) In one series of tests, the admixtures were evaluated in accordance with ASTM C 666, Procedure A (1997a), while a second series of tests evaluated the effect of the chemicals on the air void system according to ASTM C 231 (1997b) and C 457 (1998a). Table 11 shows the results of the first series of tests. As can be seen, the commercial off-the-shelf admixture passed the durability test at the normal dosage but not at the high dosage. The prototype admixture, on the other hand, did well at both dosages. This is interesting because our off-the-shelf chemicals are similar to those used in CPAR. At the time, the feeling was that the poor showing of the off-the-shelf admixture at high dosage in the CPAR study was caused by a poor bubble spacing factor and not by adverse chemical interactions between the admixtures and the concrete.

Table 11. Durability factors for concrete specimens made with a commercial admixture and a prototype admixture compared to control concrete. All specimens contained entrained air. The low dosage is typical to some concrete mixtures today while the high dosage is above normal levels and sufficient to depress the freezing point of the mixing water to -5°C.

Specimen	Dosage				
	None	Low	High		
Control	99	<del>-</del>			
Commercial	_	99	Failed		
Prototype	_	98	96		

The second series of tests under CPAR evaluated in more detail the interaction of chemicals and entrained air bubbles. The spacing factor, the average chord length, the number of voids per centimeter, the specific surface, and the paste content were determined on hardened concrete specimens representing four mixtures. The control specimens were cured in a 22°C room, while the antifreeze specimens were cured in a -7°C room. Table 12 shows air contents measured from each concrete. Mixture 1 represents a control concrete. Mixture 2 contains the same ingredients as mixture 1, except for a high dose of a commercial admixture. Similarly, mixtures 3 and 4 each contain a different prototype admixture at high dosage. As can be seen, the admixtures did not adversely affect the air content of fresh concrete.

The total air content and parameters of the air-void systems in the hardened concretes are presented in Table 13. The concretes in this table correspond to the fresh mixtures given in Table 12 after they had matured 28 days. ACI 201 (2001) and ASTM C 457

(1998a) recommend that total air content be between 4.5 and 7.5%, that the specific surface be greater than 24 (1/mm), and that the average spacing factor be 200 µm or less. The results indicate that mixtures 1 and 2 meet the required air content. (Interestingly, mixtures 2 and 4 had a higher air content in the hardened state as opposed to the fresh state.) Mixture 3 is slightly short of meeting the minimum air content, while mixture 4 had an excess of air. Too much air may weaken the concrete, though it does not harm its durability. The total air content in mixture 3 may be low, but its spacing factor and specific surface are within recommendations, which indicate an abundance of very small voids. This concrete, as well as all the others, was judged frost durable.

Table 12. Air content in fresh concrete. The admixtures were used at dosages sufficient to depress the freezing point of the concrete to -5°C. For the commercial admixture, this dosage was higher that normally used in practice.

Mixture	Content	Air content (%)
1	Control	6.0
2	Commercial Admixture	6.6
3	Prototype 1 Admixture	5.9
4	Prototype 2 Admixture	5.8

Table 13. Air-void parameters in hardened concrete containing antifreeze admixtures. These mixtures correspond to those in Table 12.

Air-void parameters	Mixture 1	Mixture 2	Mixture 3	Mixture 4
Air content (%)	5.5	7.5	4.4	8.5
Specific surface (1/mm)	31.9	29.8	36.9	26.1
Spacing factor (μm)	152	137	145	140

The air parameters for fresh and hardened concrete are important for gauging its freeze—thaw durability. The results from the CPAR study show that high doses of admixtures need not adversely affect the entrained air, nor the freeze—thaw durability of concrete, which agrees with our current studies of off-the-shelf admixtures.

Conclusions. The results from the durability tests support the idea that admixtures in high dosages do not adversely affect the concrete. This is seen in the first and second trials with the W.R. Grace products and further supported by the testing done with the Master Builders mixtures. However, the admixtures may make it more difficult to entrain air into concrete. This is seen in the scattered results from the second set of W.R. Grace concretes, where some beams were not as resistant to freeze—thaw cycling as were others. Air entraining of concrete has always been fairly sensitive and there are a number of variables that dictate what the final air content of the mixture will be. As is normal practice today for all chemical admixtures, trial batches of concrete made with the admixtures

used in this study are recommended before use in field applications. There is no reason to believe that proper amounts of air and bubble spacing factors cannot be achieved with these products.

### Time of Setting

Measurement of the time of setting by penetration resistance (ASTM C 403/C 403M [1999b]) was performed on prepared mortars based on the antifreeze concrete mixtures. These mortars were designed to simulate the mortar fraction of the concrete mixtures by calculating paste thickness on aggregate particles after all void spaces were theoretically filled.

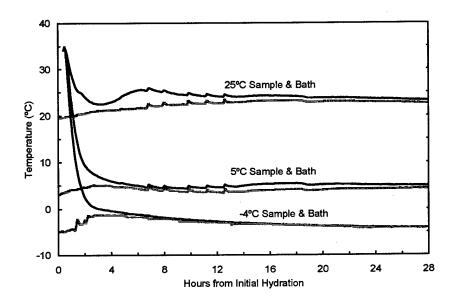


Figure 14. Typical temperature history for set time samples and baths.

We mixed 10 L of each mortar, without air entraining admixtures, at room temperature in a 28.4-L paddle mixer following ASTM C 305 (1999c). It was equally distributed among three cylindrical 160- ×175-mm plastic sample buckets with a thermocouple embedded at the center of mass in each to record its temperature during the test (Fig. 14). The freezing point of each mortar was also measured to determine the minimum temperature that it could be subjected to without freezing. Knowing both the freezing point and the thermal history of the samples assured that all penetration tests were performed on unfrozen mortar.

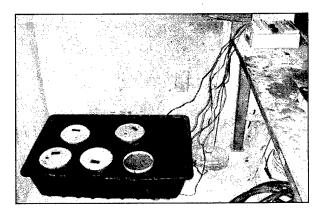


Figure 15. Samples for time of setting test in water bath. A data logger records the temperatures from thermocouples embedded into each sample.

The samples were consolidated on a vibrating table, covered with lids, and placed in water baths (Fig. 15) at each of the three test temperatures (salt water solution was used in the -4°C bath). The water level in the baths was kept above the top surface of the mortar, but below the lip of the sample container, and care was taken to avoid getting any bath water in the sample containers. The resulting initial set times for our antifreeze suites are presented in Table 14.

Table 14. Summary of initial set times.

		Set time	(hh:mm)	
			-4°C	
Admixture suite	25°C	5°C		% of 5°C control
WRG I	2:57	5:53	11:38	102
WRG II	3:12	6:23	14:28	127
WRG III	2:57	5:40	11:51	104
WRG IV			24:33	216
Control	4:34	11:21	N/A - (Freeze)	
Control	4:38	12:18**	N/A – (Freeze)	
MB I	3:47	9:53	13:57	113
MB II	5:02	15:02	25:10	205
MB III	3:37	10:46	11:50	96
MB IV	3:19	4:20	6:20	52

<sup>\*</sup> Corresponding WRG trial baseline

<sup>\*\*</sup> Corresponding MB trial baseline

Both WRG I and III came very close to meeting our original goal of setting in the -4°C bath as fast as plain mortar did at 5°C. Both admixture formulations contained the highest concentration of accelerating admixtures of the four WRG formulations, and no retarding admixtures. It was interesting to see that the shrinkage-reducing admixture contained in WRG III did not delay the time of setting as we thought it might. Not too far behind, WRG II, containing the "set neutral" DCI® S corrosion inhibitor, took a little more than 3 hours longer than the control to reach initial set. Finally, the WRG IV mixture, which contained a moderate dose of retarder, took more than double the time of the control. Because rapid setting and quick turn-around were goals in this project, the WRG IV mixture was not considered a candidate for field testing. However, concrete with retarded strength development may be very useful for bridge deck construction where early strength gain is not desirable. It is best if strength does not begin to develop until the entire deck is placed, which can take hours. More work is needed to evaluate this possibility and develop an admixture suite suitable for this application.

In a separate round of testing Master Builders suites versus the common control mixture, both MB III and IV met the goal of reaching initial set faster than the control. In fact, the MB IV mixture set in almost *half* the time of the 5°C control benchmark—proving to be the fastest setting antifreeze mixture in this study. Despite its ability to set quickly under very cold conditions, the MB IV mixture still had a reasonable setting time of just over 3 ¼ hours at room temperature. Subsequent field trials with this suite confirmed that the concrete remained sufficiently workable during placement and finishing operations, but set up quickly thereafter. Since MB IV used a different accelerating admixture than the other three suites, this product should be incorporated into antifreeze concrete applications where fast setting times are important.

MB I almost met the original goal, setting in just under 14 hours compared to about 12 hours for the control. Curiously, the MB III mixture, containing a 1% dose of shrinkage reducing admixture, set up faster than the MB I mixture. Both had the same level of accelerating admixtures and resulted in the same final w/c ratio. This result contrasted with the WRG III shrinkage reducer suite, which did not affect setting times markedly compared to WRG I. Further study is needed to determine if the faster set seen in MB III is a significant result, or only a statistical outlier encountered in this trial.

Finally, the MB II mixture, containing retarding admixture, took more than double the time of our baseline and almost four times more than MB IV. The slow setting performance of MB II was similar to WRG IV, which also contained a significant dose of retarder. These suites have the potential to be developed further for applications where delayed set *is* desired, such as bridge deck placements. Though these mixtures may set slowly, recall from the *Compressive Strength* section that the 3-, 7-, 14-, and 28-day strengths were comparable to the fastest setting antifreeze suites. This suggests that even

antifreeze concrete that remains plastic for significant periods of time could still allow prompt removal of formwork and temporary supports.

Overall, as we learned later during field tests, achieving rapid setting times at below-freezing temperatures was not as important as originally thought. Typically, the in-place temperature of the concrete placed in the field remained significantly above freezing during the period that setting took place.

Also, experience throughout our testing showed that the antifreeze concrete mixtures did not bleed like normal concrete. This allowed the concrete to be finished almost immediately following placement, unlike regular concrete, where laborers must wait until bleeding subsides. In effect, this made the time of initial set almost a moot point for the practical field use of our antifreeze concrete, but it is included here for evaluation against our original requirement.

### Critical Maturity

A final laboratory test was performed to determine if the antifreeze suites dosed into the concrete provided any additional protection from freezing damage below the design temperature of -5°C. In the field, mixture temperatures of the concrete were usually between 5 and 10°C when freshly mixed using unheated aggregates and cold water. Generally, the thermal mass of the concrete, the heat generated within the concrete during hydration, and the insulating value of the forms (or even the adjacent concrete itself) helped to keep the concrete warm for many hours despite much colder ambient conditions. Consequently, the time that it takes for freshly placed concrete to fall close to the -5°C freezing point in field placements can be substantial. The degree to which the concrete gains maturity, and thus some measure of strength, during this period helps to reduce the amount of liquid water in the concrete and, in turn, protects the concrete from damage at temperatures below its design freezing point. The field trial where in-place concrete temperatures approached the -5°C freezing point most quickly was in Concord, New Hampshire, where we encountered the coldest weather conditions. The coldest location in the structure took about 42 hours to dip to -5°C, during which time it accumulated about 255 °C-hr of maturity§.

In the laboratory, the goal was to determine how much maturity and strength the concrete needed to gain before it could survive temperatures below -5°C. The WRG I mixture was picked as a representative antifreeze suite to test. The concrete was mixed at room temperature and cast into cylinders following the mixing procedure in Appendix C, up to the 8-minute mark. Thirty minutes after water, cement, and admixtures were com-

<sup>§</sup> Computed using the time-temperature factor maturity method, with a datum temperature of -7°C commencing once all admixtures were dosed. Refer to Appendix D for a full description of using the maturity method with antifreeze concrete.

bined, sets of samples were placed in -5, -10, and -20° C coldrooms and cured for 24 hours. These were called the ½ hour group of samples. At 2-hour intervals, additional samples were placed into each of the three coldrooms. After curing for 24 hours, each set was removed from the coldroom and allowed to cure at room temperature (~28°C) an additional 7 days. At that point the samples were demolded and tested for compressive strength according to ASTM C 39 (2001b) and ASTM C 1231 (2000b). The results were compared to samples continuously cured at room temperature for 7 days.

The Nurse-Saul maturity for each set of samples, measured from the time water, cement, and admixtures combined until they were placed into the coldrooms, was determined. Any maturity gained in the coldrooms was ignored. The maturities ranged from 18°C-hr for the ½ hour samples to 260°C-hr for the 6½ hour samples. A maturity curve was also developed for early age compressive strength from room temperature samples. The relationship shown in Figure 16 was used to estimate the compressive strength that each set of samples had obtained before they were placed in the coldrooms.

The overall results of this test are presented in Figure 17. As expected, the ½ hour samples showed no damage when held at the -5°C condition for 24 hours before being cured for 7 days at room temperature. However, at -10 and -20°C, they did not recover fully; they developed only 60 to 65% of their strength relative to the samples cured at room temperature for 7 days. Note that it did not matter how low the temperature was, the degree of damage was the same. This was borne out by other preliminary tests similar to this one. No matter what temperature the fresh concrete was exposed to, or which admixture suite used, the concrete consistently showed a 30 to 40% strength reduction when held at low temperature for 24 hours. (Past studies have shown that when antifreeze concrete is held at low temperature for 28 days, it sometimes recovers full potential strength. We are not sure why this is.)

The remaining groups of samples—held  $2\frac{1}{2}$ ,  $4\frac{1}{2}$ , and  $6\frac{1}{2}$  hours at room temperature—showed no reduction in strength for any of the three freezing temperatures. So after  $2\frac{1}{2}$  hours at room temperature, which correlates to  $90^{\circ}$ C-hr of maturity, we can expect the antifreeze concretes in this study to resist an overnight freeze as low as  $-20^{\circ}$ C. The Concord job, mentioned above, reached this maturity within 9 hours at the coldest instrumented location. At that time, the internal temperature of the concrete at all instrumented locations still held between 1 and  $5^{\circ}$ C, which were well above the  $-5^{\circ}$ C freezing point. Clearly, our antifreeze concretes can resist very cold weather soon after they are placed.

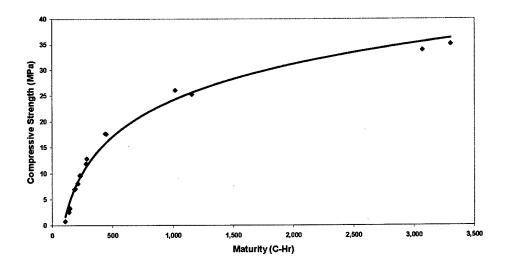


Figure 16. Relationship between strength and maturity developed from room-temperature samples.

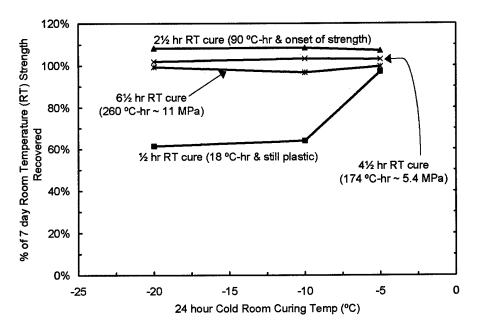


Figure 17. Recovery of compressive strength with varying room temperature curing time before exposure to different freezing temperatures.

# **Summary of Laboratory Testing and Conclusions**

The laboratory studies developed eight potential admixture suites for application as antifreeze admixtures for concrete. According to Figure 4, the concretes made with these admixtures had to satisfy requirements in seven areas:

### Workability

The rate at which concrete stiffens, or sets, is important to construction because stiffness determines the workability of the concrete, that is, whether it can be properly placed, consolidated and finished. We determined that each suite of admixtures could be used to make acceptable concrete—that could be transported up to 45 minutes from the readymix plant to the jobsite, dosed with admixtures (if needed), and worked with for up to 30 minutes thereafter.

#### Entrained Air

The resistance of hardened concrete to cycles of freezing and thawing in a moist environment is significantly enhanced when air bubbles are intentionally incorporated into the fresh concrete. We determined that concrete made with seven of the eight admixture suites developed in this study could be entrained with air by using conventional air entraining admixtures, but the work did not include an in-depth assessment of the quality of the air void system. Thus, it is recommended that trial batches be made to find the proper dosage of air-entraining admixture for each cement brand and particular mixing operation. This area needs further work.

## Initial Freezing Temperature

The first function of an antifreeze admixture is to depress the freezing point of the water in the fresh concrete. The water in normal concrete freezes at approximately -1°C. We succeeded in reducing the initial freezing point of fresh concrete to at least -5°C.

#### Compressive Strength

The second function of an antifreeze admixture is to promote strength development in concrete while its internal temperature is low. The goal was to use a sufficient amount of accelerating admixtures to force concrete held at  $-5^{\circ}$ C to gain strength at least as rapidly as normal concrete held at  $5^{\circ}$ C. We achieved that goal for concretes made with seven of the eight admixture suites.

# Freeze-Thaw Durability

The goal was to determine if the admixtures used in this study affected concrete or if they prevented air from being entrained into concrete. As the results demonstrated, the admixtures themselves do not reduce the freeze—thaw durability of concrete, nor do they prevent air from being intentionally entrained into concrete. However, the air content of antifreeze concrete was more difficult to control than it was in normal concrete. It is recommended that trial batches of concrete be made to fine-tune air-entraining admixtures into the concrete.

### Time of Setting

The rule-of-thumb is that initial setting time doubles for each 10°C in temperature (Korhonen 2002). For normal summertime construction, it is not uncommon for finishing crews to wait several hours before concrete can be finished. The goal was to create a concrete held below freezing that would set as fast as normal concrete held at 5°C. Though this was a tough goal to reach, we discovered that the antifreeze concrete, because it did not bleed, could be finished nearly immediately after placement. In addition, the internal temperature of the field concrete test sections stayed well above -5°C during the first 24 hours following placement. Thus, time of setting at below freezing was not a useful measurement in this study. Nevertheless, several of our antifreeze concretes set fairly rapidly just the same.

#### Critical Maturity

In practice, it is unlikely that freshly placed concrete will cool off to the  $-5^{\circ}$ C initial freezing point of the concrete until several hours have passed. In fact, our field experience shows that concrete structures can reasonably be expected to maintain their internal temperature well above this value for at least 24 hours, even under harsh conditions. Laboratory testing showed that fresh antifreeze concrete could not immediately withstand temperatures below its initial freezing point without damage. However, with minimal maturity gain and the onset of compressive strength, the concrete could withstand internal temperatures as low as  $-20^{\circ}$ C (see Fig. 17). As a rule of thumb, if the concrete starts out at a temperature of at least  $10^{\circ}$ C and stays above  $0^{\circ}$ C for at least 6 hours, it will be able to resist frost damage from one temperature excursion to  $-20^{\circ}$ C (concrete temperature).

### 3 FIELD EXPERIMENTS

#### Introduction

A critical part of this study was to achieve the reliable use of antifreeze-protected concrete in construction of transportation facilities and other structures. This required assurance that formulations and tests work for full size batches under actual construction conditions in subfreezing weather. The study needed to address the effects of antifreeze-protected concrete compared with the temperate alternative with regard to field-specific issues, including:

- Batch mixing of formulations in full-scale plants.
- Transportability of mixtures.
- Emplaceability of mixtures.
- Labor, equipment, and material cost penalties, if any.
- Compatibility of winter concrete emplacement with the construction process.
- Cleanup of equipment.
- Quality assurance\*\*.

These matters were explored in five full-scale field projects shown in Table 15. Detailed descriptions of each test follow.

Location	Date	Volume (m³)	Suite	Description
Littleton, NH	10-Dec-01	2.0	WRGII	Bridge curbing
Rhinelander, Wl	27-Feb-02	4.5	WRG I & II	Pavement section
North Woodstock, NH	12-Dec-02	3.0	MB IV	Footing
West Lebanon, NH	18-Dec-02	7.0	MB IV	Bridge curbing
Concord, NH	14-Feb-03	5.5	MB IV	Sidewalk

Table 15. Field studies.

## Littleton, New Hampshire

A traffic accident created the first opportunity to field test one of the antifreeze concretes developed in the laboratory. In mid-October 2001, a northbound tractor-trailer tipped over and slid into a downhill curve on Interstate 93 near Littleton, New Hamp-

<sup>\*\*</sup> Development and verification of the Initial Freezing Point and Maturity measurement methods used for QA/QC in the field are detailed in Appendices C and D.

shire. The truck, lying on its driver's side, caromed off the guardrail of bridge 190/058 and proceeded to slide down hill—a 3% slope—another 100 m before coming to rest. Fortunately, no serious injuries were reported and the only damage recorded was to the truck (Fig. 18) and to a 12-m section of bridge curbing (Fig. 19). The Bureau of Bridge Maintenance, NHDOT, offered the damaged curbing as a test site for our concrete. This was both a practical and timely application for this technology.

Nice weather, strange as it may sound, delayed this test from 26 November to 10 December. The weather was too mild between those dates to qualify as "cold." In fact, the first 5 days of December were 10°C above normal, where normal usually hovers around the freezing mark. However, the weekend prior to 10 December was perfect—around freezing during the daytime and 10°C lower at night. The outdoor air temperatures for the day of the pour, as well as the following 7 days, are shown in Figure 20.



Figure 18. Truck slid downhill on its side into bridge curb (17 October 2001).



Figure 19. A 12-m section of guardrail was pushed up and back from the curb.

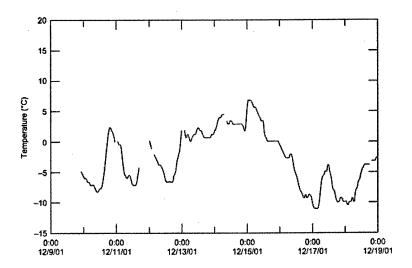


Figure 20. Weather data from St. Johnsbury, Vermont (NOAA 2001).

### The Damaged Section

The area of curbing that needed to be repaired measured approximately 43 cm wide by 25 cm deep by 12 m long (Fig. 21). It contained steel reinforcing bars, with anchor bolts for the guardrail welded to the bars. The repair was instrumented at three locations (near each end and at the middle of the 12-m length) with thermocouples at three depths: 13 mm below the finished surface, at the center of mass, and 13 mm up from the bottom of the concrete. The thermocouples were attached to dataloggers set to record at ½-hour increments (Fig. 22).

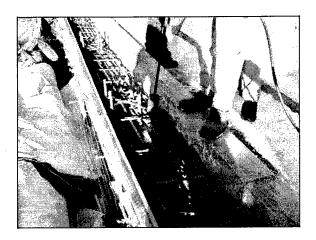


Figure 21. Torching away ice.



Figure 22. Initializing dataloggers.

#### The Mixture

The ready-mix plant was located in St. Johnsbury, Vermont, about 30 km west of Littleton. At approximately 0855 on 10 December 2001, some of the mixing water (8°C), all of the aggregate (with air-entraining admixture ribboned onto the sand), and the Type II cement were loaded into the concrete truck's drum. The access to the truck's drum was washed down and the drum was turned for a few minutes (washing down the drum can add an extra few gallons of water into the mixture). With the drum stopped, the first admixture was pumped into the front part of the drum (Fig. 23). Immediately following that addition, a mid-range water reducer was poured onto the concrete near the top of the drum to keep either admixture from directly contacting the other. The drum was turned at mixing speed for about 1 minute. It was stopped briefly while the rest of the mixing water and a high-range water reducer were added into the drum, then mixing continued. The w/c ratio of the mix at this point was expected to be 0.40 and its slump was estimated (by observing the mobility of the concrete inside the drum as it slowly turned) to be between 180 and 200 mm.

The truck departed the ready mix plant at 0914 and arrived at the bridge at 0935. The slump was estimated to be about 100 to 130 mm (it was expected to be around 70 mm). At 0950 the second part of the antifreeze admixture was added by pails into the drum along with about 2.5 kg of sand sprinkled with air-entraining admixture (Fig. 24). The expected w/c ratio, slump, and freezing point of the concrete were 0.45, 165 mm, and -5.3°C, respectively, including these jobsite additions (these measurements were obtained when the mixture temperature was 10°C). After 30 revolutions of the drum, the slump was estimated to be about 200 to 220 mm. Because the repair area sloped at 3%, the decision was made to wait until the slump dropped to around 150 mm. At 1014, the slump had not changed perceptively and the temperature of the mixture was 8°C, measured with a hand-held thermometer. By 1024 the concrete appeared to have stiffened

somewhat. Four minutes later, at 1028, the slump was estimated at 180 mm and falling, and the mixture temperature was 7°C.

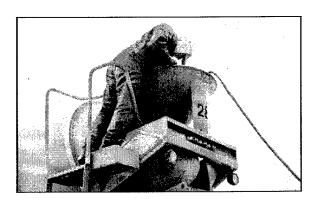


Figure 23. Pumping first admixture into drum at batching plant.

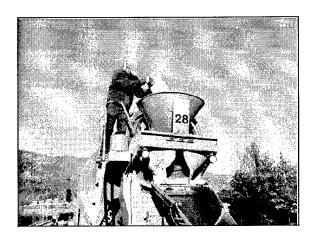


Figure 24. Adding antifreeze admixture and sand laced with air-entraining admixture on the jobsite.

# Placing the Concrete

Concrete placement began at 1035. A sample of concrete, obtained in a wheelbarrow soon after the placement started, was measured for various properties at 1040 (Fig. 25): 2310 kg/m³, 180-mm slump, 8°C, 5.1% air (the slump was quite a bit higher than expected but the temperature and air content were expected based on previous testing at St. Johnsbury). Three 51- by 102-mm plastic cylinders were also obtained at that time and

measured for their freezing point within 25 minutes, providing an average value of -5.2°C (within the accuracy of our equipment, this was pretty much what was expected).



Figure 25. Testing the concrete.

The work of placing the concrete lasted 20 minutes, ending at 1055. Emplacement consisted of very briefly consolidating the concrete with a vibrator, screeding off the concrete with a piece of lumber, and smoothing the resulting surface with a hand-held magnesium float (Fig. 26). In past testing, this concrete mixture showed no tendency to bleed. Because of this, the consolidation, screeding, and floating could be done immediately following the chute of the truck. The entire finishing operation was completed by 1115. The finished concrete was covered with a sheet of plastic (to minimize evaporation) and with a blanket of insulation.

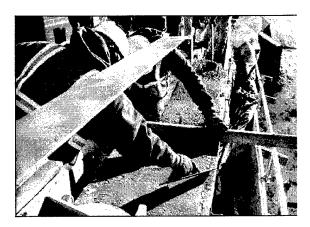


Figure 26. Placing, consolidating, screeding and floating the concrete.

# Strength Gain

Based on a maturity curve (Nurse-Saul) developed in the laboratory (Fig. 27), and on the temperatures that were recorded from the concrete as it cured, it was estimated that the concrete had developed approximately 7 MPa compressive strength by 2400 (12 hours) and in excess of 17 MPa by 1200 the following day (Fig. 28). The design strength of this concrete was about 35 MPa.

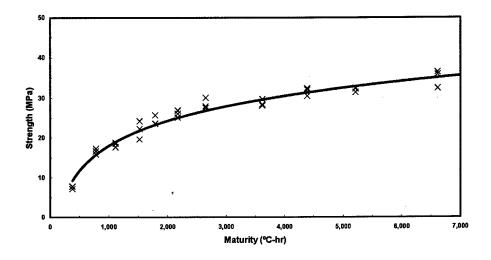


Figure 27. Maturity curve developed in the laboratory.

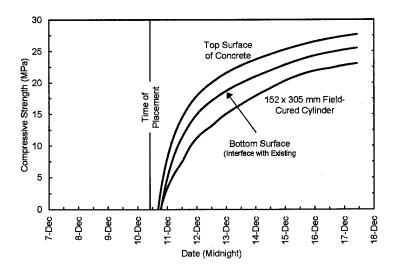


Figure 28. Estimated strength development using maturity method.

#### Relative Costs

If this work were to be done using normal procedures, it may have been necessary to build a temporary heated shelter around the repair area, such as shown in Figure 29. To build such a shelter, heat it for 1 day before placing concrete, and then heat it for 5 more days while the concrete cures, followed by dismantling, could cost up to \$2500 (Corliss 2001). In comparison, the only extra cost of using the admixtures used in this test was the cost of the admixtures themselves—no other protection was required, other than the sheet of plastic. We estimate the admixtures to increase the cost of the 2 m³ of concrete ordered by less than \$250. The cost of the concrete itself was around \$190.

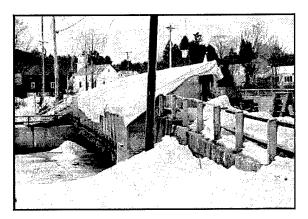


Figure 29. Typical temporary protection for bridge curbing repair.

#### Lessons Learned

Wash Water. Usually, the fins in the drum of the concrete truck are washed down before the truck leaves the ready-mix plant. This process was allowed on this job. We believe that this was one source of extra water that contributed to a higher than desired slump. Perhaps this process should be avoided on future winter projects, or the wash water should be accounted for. Another possible explanation for the increased slump was the fact that the concrete was somewhat cooler during actual placement (7°C) in Littleton compared to what it was during preliminary testing (10°C) in St. Johnsbury.

Bleed Water. The concrete, though of very high slump, was ready for finishing very soon after placement because the concrete did not bleed.

Maturity Curve. A maturity curve developed in the laboratory was very helpful in determining when the concrete had cured sufficiently to open it to service loads. The maturity curve did require several days of testing prior to placing the job concrete.

Freezing Point Determination. Though the freezing point of the fresh concrete was not determined until after the concrete was placed, it nonetheless indicated that, despite the concrete having more water in it than expected, it met our specifications.

Cost. Though admixtures can double the cost of each cubic meter of concrete, the resulting antifreeze concrete can be substantially less expensive in emplaced costs compared to conventional, thermally protected concrete.

#### Rhinelander, Wisconsin

The intersection of routes 8 and 17 in Rhinelander, Wisconsin, was the location for the second field test of antifreeze concrete. Unlike the situation for the first field test in New Hampshire, where early strength development was not so critical, the emphasis this time was on opening the road to traffic within 48 hours. Adding to the challenge, the weather was also much colder this time around.

### The Repair Section

The area of concrete to be replaced measured approximately 3 m wide by 7 m long by 250 mm deep. At approximately 0900 on 27 February, the section was removed (Fig. 30) and holes for dowel bars were drilled (Fig. 31) into surrounding concrete. (The section was saw cut around its perimeter in November 2001 to facilitate removal.) The section was instrumented at six locations with thermocouples at three depths: 25 mm below the finished surface, at the center of mass, and 10 mm up from the bottom of the concrete. In addition, one thermocouple monitored the temperature of the base material, approximately 20 mm below the bottom of the fresh concrete and one was used for monitoring the ambient air temperature. Figure 32 shows three of the six positions where thermocouples were placed, and the data collected from one thermocouple from each of these three locations are presented later. The additional three thermocouple positions were similarly placed at edge, corner, and center locations elsewhere in the pavement section. The thermocouple wires were attached to dataloggers set to record at ½-hour increments.

#### Moisture Control

As with all concrete mixtures, it is important to control the amount of water added into the mixture. Typically, water comes from three sources: water added during mixing, water contained within the admixtures, and free moisture on the aggregate. (The truck driver emptied all previous wash water from the truck's mixing drum and was instructed not to add additional water.) Of these three sources, water from the aggregate was the most difficult to control. Therefore, the coarse and fine aggregates were sampled from the silos and tested for moisture content about 1-½ hour before the batching process began.



Figure 30. Removing a section of concrete roadway in Rhinelander, Wisconsin.

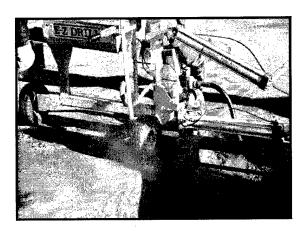


Figure 31. Drilling dowel holes.

The procedure illustrated in Table 16 was used to control the addition of water associated with the aggregates. Using the measured free moisture contents and the nominal SSD weights for aggregates in the mixture determined how much of each type should be dropped into the batcher. Because the amount of aggregate could not always be closely controlled, the amount of water actually added was computed after the real drop weights were known. Usually the amount of aggregate dropped was close to the amount desired; however, the mixing water metered into the drum was still adjusted accordingly.

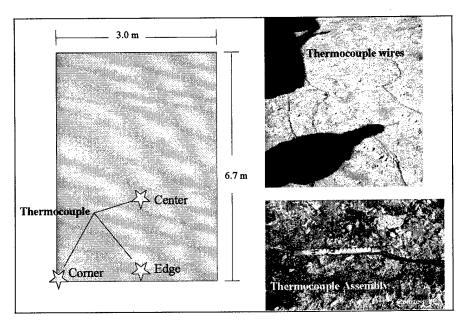


Figure 32. Instrumenting the pavement.

Table 16. Accounting	for free moisture in the a	aggregate.
Mix design	Desired	A

	Mix	design	De	Desired		ctual
Ingredient	SSD weight (kg)	Free water content (%)	Drop weight (kg)	Free water (kg)	Drop weight (kg)	Free water (kg) <sup>†</sup>
Cement	2160	_	2160	<del>-</del>	2159	
Fine aggregate	3537	6.6	3771	234	3773	234
19-mm coarse aggregate	4342	1.7	4415	74	4415	74
38-mm coarse aggregate	816	-0.4	812	-3.2	817	-3.2

<sup>\*</sup> Desired drop weight = SSD weight × (1 + free water%/100)

As it turned out, the w/c ratio of the concrete mixed for this test was higher than expected. The freezing point measurements taken from the concrete as it was being placed revealed this. A freezing point of -5.5°C was expected, but the measured value was -5.0°C. By using the relationship between admixture concentration and freezing point developed during preliminary trial tests the week earlier (Fig. 33), the w/c ratio was calculated to be around 0.49 as opposed to the expected 0.43. This excess moisture could have

<sup>&</sup>lt;sup>†</sup> Actual free water weight = actual drop weight  $\times$  [1 – {1/(1 + free water%/100)}]

resulted from the aggregate being wetter than expected, or from more water being added during the pumping operation than realized, or both.

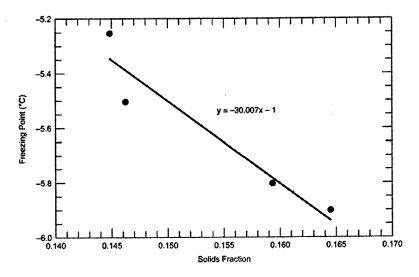


Figure 33. Relationship between amount of admixture in the mixing water and freezing point.

Although more water was inadvertently introduced into the concrete, the concrete's freezing point still met the original goal of this project: to develop a concrete that could resist freezing down to -5°C.

# The Mixture

Batching for 4.5 m³ of antifreeze concrete began at approximately 1100, 27 February 2002. The sand and coarse aggregate, stored in heated silos, were first added into the drum of the truck (heated aggregate was not necessary but that was the only aggregate available). Next, the cement (Type I, 476 kg/m³) was added followed by cold mixing water containing an air-entraining admixture at 1118. This produced a mixture temperature of approximately 10°C, our target. Higher temperatures would have caused problems with rapid slump loss. The truck mixed the ingredients for 5 minutes before departing at 1123. Normally, the top fins in the truck's drum are washed down at this point to clean off any unmixed material. This, however, tends to add a few gallons of unintended water into the drum, so washing was not permitted. The calculated w/c ratio of the mixture at this point was 0.30 and the slump was estimated to be 20 mm according to the resistance meter on the truck.

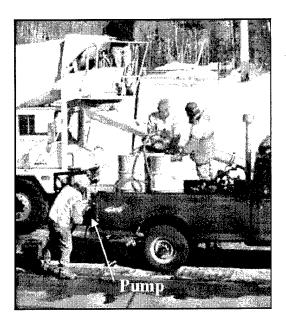


Figure 34. Pumping admixture into truck.

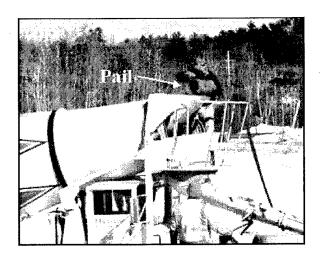


Figure 35. Adding plasticizer.

Once at the construction site, the first admixture was pumped into the truck's drum at 1135 using an ordinary gas-powered pump (Fig. 34). The drum was turned for a few minutes, stopped, and backed up, so that a mid-range water reducer could be poured into the fresh concrete at 1140 (Fig. 35). The drum was turned for another few minutes and backed up again to allow a high-range water reducer to be poured onto the fresh concrete at 1144. The drum was turned a few minutes more when the slump was estimated to be 80 mm. A higher slump was expected, so 27 L of water was added by pail into the mixer

at 1150. At 1155, the final admixture and a second dose of air-entrainer were added and mixed until 1200. The expected w/c ratio of the final mixture was 0.43.

### Placing the Concrete

Concrete placement began at 1200 by Oneida County personnel. A sample of concrete, obtained in a wheelbarrow soon after the placement began, was measured for slump (250 mm), air content (8%) and temperature (18°C) at 1203. Three 51- by 102-mm plastic cylinders were also obtained at that time and measured for freezing point within half an hour: -5.0°C. A second wheelbarrow was filled at about 1203, from which eleven 152- by 305-mm cylinders were cast. The truck completed placing concrete at 1207 and 0.5 m³ of waste was emptied from the truck at 1215. The slump of the concrete at that time was observed to be about 100 mm.

The work of placing the concrete lasted 30 minutes, ending at 1230. It was placed with the truck's chute, leveled, and compacted with a vibratory screed (Fig. 36). The resulting surface was immediately finished with a magnesium bull-float (Fig. 37) and no further finish work was done to the concrete. The surface was sprayed with a sealing compound at 1300. The finished concrete was covered at 1500 with a sheet of plastic (to minimize evaporation) and with a 25-mm-thick insulation blanket (to accelerate strength gain, but not needed for freezing protection) (Fig. 38).



Figure 36. Vibratory screed.



Figure 37. Floating.

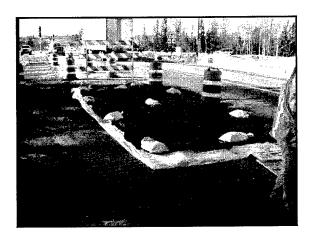


Figure 38. Insulation not needed for protection, but to hasten curing.

# Strength Gain

To estimate strength development in the pavement, the 11 samples cast during the placement operation were tested for compressive strength at various ages, while corresponding temperature data were collected from a dummy cylinder and from various points in the fresh concrete. Nine cylinders were stored next to the pavement in picnic coolers. The coolers slowed heat loss from the samples. One of these cylinders contained an embedded thermocouple from which a Nurse-Saul maturity function (ASTM 1074 [1998b]) was developed and related to the strengths determined at various ages from the cylinders. Two cylinders were stored indoors in 23°C water and were assumed to remain

at that temperature until testing a day later. All specimens remained in their plastic molds until testing (Fig. 39). The maturity curve (Fig. 40) was developed from both the strength data from the samples stored in the picnic coolers and in the water bath along with the temperature data obtained from the dummy cylinder. Once the maturity curve was developed, the pavement's strength could be estimated for any location by using the temperature-time history recorded from it (Fig. 41).

The resulting strength gain curves for three critical points in the pavement are shown in Figure 42. The central portion of the pavement reached nearly 20 MPa within 24 hours. At the 48-hour mark, that same section of concrete attained 29 MPa, the area 10 mm inside the edge attained 21 MPa, and the corner attained 15 MPa. The corner reading is considered to be conservative as the thermocouple at that location was pressed up against the existing concrete. It, therefore, recorded the combined temperature of the fresh concrete and the existing hardened concrete. A truer measure of the corner concrete would have been obtained by positioning the thermocouple 10 mm away from the existing concrete, as was done for the edge reading. The concrete in the corner was most likely stronger than shown.



Figure 39. Developing the maturity curve.

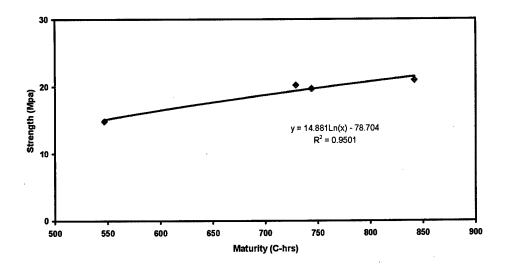


Figure 40. Relationship between strength and the time-temperature history.

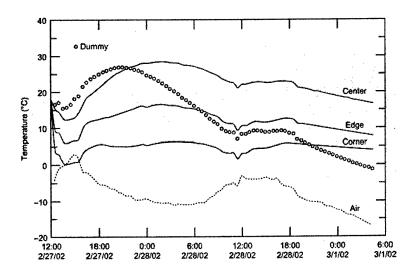


Figure 41. Temperature history over 42 hours.

Though not shown in Figure 42, the pavement began to develop considerable strength at a very early age. For example, the pavement was strong enough to stand on 2 ½ hours after the concrete was placed (Fig. 43). Note that the strength-maturity relationship developed in Figure 40 was based on data from 11 to 20 MPa. Therefore, it was valid for predicting strengths near the project's target of 21 MPa, but not at very low strengths and ages. Additional data from samples tested at lower maturities could easily solve this matter if low strength estimates were necessary.

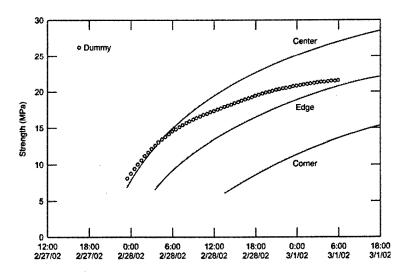


Figure 42. Strength vs. time based on Figures 40 and 41.



Figure 43. Standing on the concrete 2-1/2 hours after placement.

It is interesting to note that the strength of the field-cured cylinders does not represent the strength as it developed in the pavement. As shown in Figure 42, the cylinders (dummy) started out by overestimating strength, by being warmer than the pavement at the start, and then by underestimating the central region of the slab as they cooled off. The cylinder strengths provided little correlation with strength near the pavement's edge.

# Opening to Traffic

The insulation blanket was removed from the road 1030, 1 March 2002, when the air temperature was  $-11^{\circ}$ C. The new concrete was opened to traffic  $2^{-1/2}$  hours later at 1300,

when the air temperature was -9°C. No cracks or other signs of distress were noted. The concrete was opened to traffic approximately 48 hours after the concrete was placed, meeting the deadline originally set.

#### Relative Costs

Cold weather can more than double the cost of concreting. For example, concrete costing \$98/m³ can cost another \$98 in labor to emplace during the summer, depending on the structure being formed. When winter sets in, the in-place cost of concrete can easily increase to \$400/m³ because of the heat, temporary enclosures, and insulation needed to protect it from freezing. For this work, it was estimated, based on admixture rates in New Hampshire, that the cost of the concrete was increased by only \$130/m³. Thus, the in-place cost of this antifreeze concrete could be much less than using conventional winter concreting techniques. For pavement work, such as was done in this project, it is likely that the concrete would not have been placed had it not been for the antifreeze formulation's ability to reduce costs closer to the summertime level.

### Lessons Learned

Moisture Control. It was difficult to control the w/c ratio. In this study, extra water got into the mixture—most likely from snow that had fallen on the aggregate piles the night before and from the water used to prime the admixture pump.

Freezing Point Measurements. It was important to measure the freezing point of the fresh concrete so that the actual w/c ratio could be estimated. This measurement also determined the level of freeze protection finally in the concrete.

Speed of Determining the Freezing Point. The freezing point was measured within ½ hour after placement began. Ideally, it would be good to determine that before placement, so that extra admixture could be added if needed, the concrete could be further protected thermally if conditions actually warranted, or the concrete could be rejected. Work needs to be done to automate freezing point measurements.

Finishing. A magnesium float seemed to tear the concrete surface. A steel float seemed to work much better. This should be investigated further, as steel floating normal concrete has been accused of sealing the surface and causing blisters. However, because this concrete, when properly proportioned, does not bleed, a steel float may not cause the same blistering problems.

### **Epilogue**

We inspected the concrete surface in August 2003. It showed no signs of deterioration. However, there was a fine network of shallow map cracks in the surface of the concrete. These could only be detected by sprinkling water on the concrete. This was likely caused by too high a w/c ratio. Recall that the in-place w/c was 0.49 when it should have been 0.43.

# North Woodstock, New Hampshire

The Gordon Pond Brook Bridge on New Hampshire Route 112, 1 km west of North Woodstock, New Hampshire, was being widened during the winter of 2002–2003 (Fig. 44). We used this opportunity to develop a concrete mixture for casting one of the new footings needed for the widening. This job represented the third winter concrete test undertaken in this FHWA pooled-fund project.

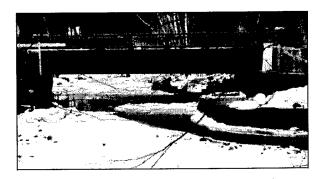


Figure 44. North Woodstock, New Hampshire, 12 December 2002.

### The Repair Section

The footing measured approximately 1 m wide by 2 m long by 1 m deep, and it contained steel reinforcing bars, including some that protruded out from the surface of the final grade (Fig. 45). Because it was a new footing to be placed on the stream bottom, a cofferdam was built around the footing and pumped dry to expose the streambed so construction could proceed (Fig. 46).



Figure 45. Footing form and exposed rebar.

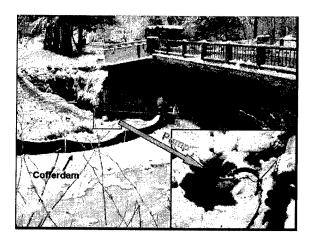


Figure 46. Cofferdam facilitated work below stream level.

### The Mixture

The ready-mix plant was located about 25 km south of the bridge in Campton, New Hampshire. The approximate driving time from the plant to the bridge was 20 minutes. Because a trial batch of the concrete made on 4 December 2002 showed that the concrete dropped from an initial slump of 175 mm to 100 mm in 20 minutes, it was decided that all admixtures would be pumped into the truck at the jobsite to allow for reasonable working time (we also used this procedure in Wisconsin with some success). Thus, on 12 December 2002, the truck was loaded at 1115 with the cement (392 kg/m³ Type II), sand, coarse aggregate, air entrainer, and water (cold) to make 3 m³ of concrete with an expected w/c ratio of 0.25, including the water in the aggregate (moisture contents were obtained from the aggregate piles 1 hour before batching the concrete). The drum was

turned at mixing speed for 3 minutes and then stopped before the truck departed for North Woodstock. It snowed heavily the night of 11 December, so special care with loading aggregate stocks was taken to avoid dispensing snow into the mixing truck. As we will discuss later, however, quite a bit of this extra moisture must have gotten into the batch.

Once the truck reached the jobsite, the first admixture was pumped into the truck between 1152 and 1157. The drum was then turned at mixing speed for 3 minutes. A plasticizer was added at 1200 directly onto the concrete (the drum was backed up to facilitate this). The drum was again turned for 3 minutes, and the final admixture was pumped into the truck between 1205 and 1210. The concrete was mixed a final time for 3 more minutes and then discharged into the footing form beginning at 1215.

# Placing the Concrete

Concrete placement began at 1215 (Fig. 47). A sample of concrete was obtained in a wheelbarrow at 1216. Soon after that, the fresh concrete was tested and found to have a temperature of 9°C, an air content of 10% (high), a unit weight of 2240 kg/m<sup>3</sup>, and a slump of 200 mm (the concrete was cohesive, showing no tendency to segregate). Three 51- by 102-mm plastic cylinders were also obtained at that time and measured for a freezing point (Fig. 48) of -4.4°C (our target was -5.0°C). This suggested that the water content was higher than expected—most likely caused by snow getting into the aggregate when it was taken from the piles. Further analysis showed that the actual w/c ratio of the concrete, instead of being 0.37, was 0.50. (This explains why the freezing point did not meet expectations, and probably why the air content was so high. Typically, when low w/c ratio concrete is dosed with extra water, the air content increases.) The concrete placement took approximately 20 minutes, and another 15 minutes were needed for finishing operations. Rebar protruded from the final surface, so the concrete was covered with an insulation blanket at 1450. The insulation was not necessary to thermally protect the mass of the concrete but to prevent the protruding rebar from causing local freezing as the air temperature dropped.

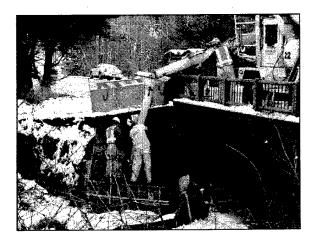


Figure 47. Placement operations in progress.

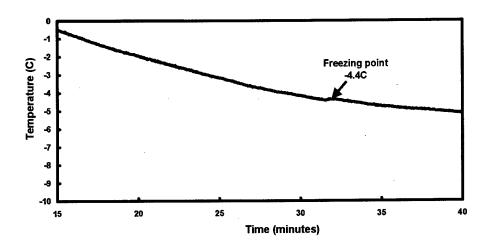


Figure 48. Though not as strong a signal as previous tests, the freezing point of fresh concrete was estimated to be approximately –4.4°C.

# Strength Gain

To estimate the compressive strength development of the concrete, we cast samples with material taken from the truck during the placement operation: 21 cylinders were stored next to the bridge in picnic coolers, 9 cylinders were stored in the stream, and 20 were stored at room temperature. Periodically, cylinders were transferred to CRREL for strength testing. All specimens remained in their plastic molds until testing, and all cold samples were warmed to 5°C before breaking to ensure that the cylinders were not frozen. A maturity curve was developed with the strength data from the samples stored in the

picnic coolers, in the stream, and at room temperature using temperature data obtained from instrumented dummy cylinders from the three locations. Once the maturity curve was developed, the footing's strength could be estimated for any thermocouple location in the structure by using the temperature/time history recorded from it.

The resulting strength gain curves for three critical points in the placement are shown in Figure 49. The center of the footing reached 20 MPa in approximately 2 days, while the concrete 75 mm inboard of the plywood forms attained that same strength a bit slower, taking about 3 days. The coolest portion of the footing, that next to the existing bridge wall, required almost 4 days. No portion of concrete was ever in danger of freezing. Because the water temperature was quite low—about 1°C—the forms were not removed until the concrete cooled to a temperature that would not cause thermal cracking when exposed to the water. The forms were in place for at least 7 days.

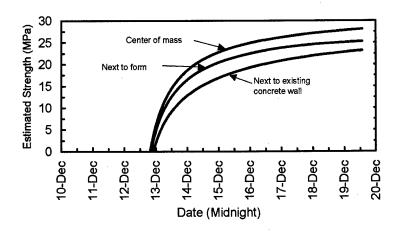


Figure 49. Strength gain curves for three locations in the footing.

### **Temperatures**

Figures 50 and 51 show the temperatures of the outdoor air and three locations within the concrete. Notice that the air during the days prior to this project was relatively cold, but during the job, and for several days thereafter, it remained around the freezing mark. This certainly did not present any challenge to the concrete, as witnessed by its in-place temperatures for this casting of considerable thermal mass. The weather could have been significantly colder.

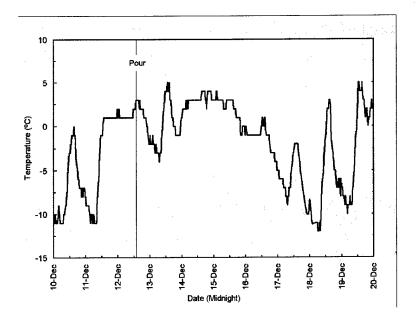


Figure 50. Ambient air temperatures at Laconia, New Hampshire—80 km away (NOAA 2002a).

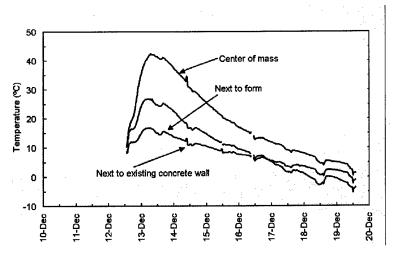


Figure 51. Concrete temperatures.

### Lessons Learned

Site Addition of Admixtures. This proved to be trouble-free. Each admixture required about 5 minutes to be pumped into the truck. More importantly, adding the admixtures at the site assured that slump loss would not be a problem. The mixture remained workable

for nearly 60 minutes. The higher than expected w/c ratio probably contributed to the long period of workability.

Snow Added to the Water Content in the Mixture. A similar problem occurred with our second field test in Wisconsin the previous winter. There, snow-covered aggregate was placed into heated silos the night before the job started. Though moisture samples were obtained from the bottom of the silo the next day, more water got into the mixture than anticipated. One solution for better moisture control is to use extra care when loading silos so that snow and ice are minimized. Another solution is to develop a method to measure the freezing point of the fresh concrete while it is still in the truck. Currently, we use dry ice and dataloggers to make this measurement within 20 to 30 minutes. Work should be undertaken to automate this process to make it faster and more practical for field use.

The Air Content Was Too High. The trial batch prepared on 4 December showed a high air content as well: 12%. The air-entraining admixture dosage was lowered for this mixture, but obviously not enough. The solution would be to decrease the admixture even more. However, as noted earlier, the unexpectedly high w/c ratio probably also contributed to a high air content. Even though the air content was excessive, design strengths developed promptly.

The Weather Was Not Cold Enough. As has been the case with our other studies, it is difficult to predict how cold the weather can be before there is danger of freezing the concrete. This is becoming the weakness of our project—not being able to predict the temperature of the concrete over time. Now that we seem able to place concrete in the cold, work should begin to define how cement cures in the presence of admixtures at low temperatures so adequate models can be developed.

Working with the Concrete. It was easy to add the admixtures into the truck at the site—it took only about 10 to 15 minutes—and the resulting concrete was easy to work with and to finish.

### West Lebanon, New Hampshire

New concrete curbing and two abutments were repaired on the west side of the Trues<sup>††</sup> Brook Bridge on New Hampshire Route 12A about 5 km south of West Lebanon, New Hampshire. The repair, the fourth one undertaken in the FHWA TPF-5(003) project, used two consecutive truckloads of concrete and was carried out during the coldest weather of all the tests conducted to date. In addition, a new method of dosing and mixing the admixtures was tested and found to work quite well. The bridge carries about 5000 vehicles per day.

<sup>&</sup>lt;sup>††</sup> Officially USGS data list this stream as Blood Brook, but many refer to it as Trues Brook, so we deferred to local convention.

# The Repair Section

The area of curbing, running north and south along the west side of the bridge, that needed repair measured approximately 460 mm wide by 380 mm deep by 32 m long. It contained steel reinforcing bars, with anchor bolts for the guardrail welded to the bars (Fig. 52). The abutment repairs, on both the north and south ends of the curbing, measured 380 mm wide by 230 mm deep by 2 m long, and sloped away from the bridge at a 46% grade.

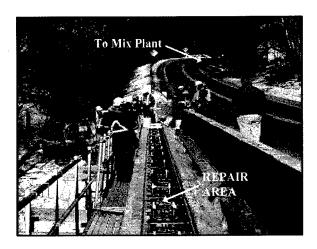


Figure 52. Trues Brook Bridge ready for repair.

### The Mixture

The ready-mix plant was located about 1.5 km north of the bridge. The first truck was loaded with the first admixture at about 0945 and then with enough cement (392 kg/m³, Type I/II), sand, coarse aggregate, air entrainer, plasticizer, and cold water to make 3.5 m³ of concrete with a w/c ratio of 0.25 (excluding the water fraction of the admixture already in the truck). The drum was turned at mixing speed for 3 minutes and stopped before the second part of the antifreeze mixture was pumped into the drum at 1005. The final w/c ratio was 0.37 (the control mixture, the basis for this mixture, had a 0.44 w/c ratio). The second admixture, unlike in previous tests where immediate mixing occurred, remained unmixed with the concrete until the truck arrived at the bridge at 1015. The mixing was delayed until the truck arrived at the jobsite to avoid any slump loss that was likely to occur during transit, as the second admixture was an accelerator. It worked! Once the concrete was mixed for 3 minutes, it came out of the truck with a full 200-mm slump. Normally, we design the mixture to start out at the ready-mix plant with a high slump (200–230 mm) so that by the time it gets to the jobsite it still retains reasonable workability (100-mm slump). By delaying the final mixing time, we essentially created a

zero-delivery-time concrete. This has interesting implications for reducing admixture dosing rates and thus costs. Once the concrete was mixed, it was discharged into the forms.

The identical mixing process occurred with the second truckload, producing the same results (except that a few balls of dry, unmixed concrete came out at the beginning of the pour). Because the plant was so close to the jobsite, there was no concern about creating a cold joint between the two consecutive placements. Consequently, the second truck was not batched until the first truck had completely discharged its load. In retrospect, the second truck could have waited onsite for a while behind the first truck without affecting the concrete onboard because of the delayed mixing process.

### Placing the Concrete

Concrete placement from the first truck began at 1025. A sample of concrete was obtained in a wheelbarrow at 1030. Soon after that, the fresh concrete was measured to have a temperature of 10°C, an air content of 11.1% (high), a unit weight of 2240 kg/m³, and a slump of 200 mm (target value). Three 51- by 102-mm sample cylinders were also obtained (Fig. 53) at that time and measured for a freezing point (Fig. 54) of -6.6°C (our target was -5.0°C or below). Numerous 76- by 152-mm concrete cylinders were also fabricated for later strength testing. The strength cylinders were used to estimate the strength gain of the concrete in the bridge using the maturity method.

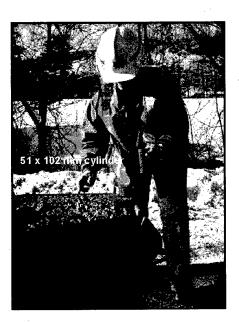


Figure 53. Obtaining freezing-point samples.

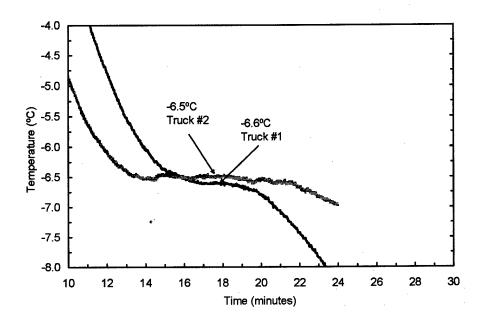


Figure 54. Freezing point measurements.

The concrete was placed on the northern abutment (wing) first (Fig. 55). It was held in place on the slope by a piece of plywood nailed to the top of the sidewall forms. Because the concrete was expected to stiffen rapidly, the piece of plywood was removed 15 minutes after the concrete was placed (Fig. 56). This allowed the fresh surface of the concrete to be finished, while avoiding the tendency of high slump material to slide off the repair area during placement. The truck discharged the last of its concrete at 1045.



Figure 55. Placement of concrete began at 1025.

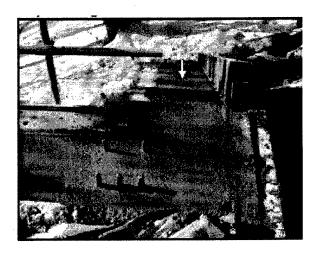


Figure 56. The top form was removed (arrow) from the abutment 15 minutes after the concrete was placed to allow finishing.

The second truck arrived at 1105, mixed for 3 minutes, and then began placing the concrete at approximately 1115. At 1120 a wheelbarrow was filled with concrete from which measurements of the fresh concrete properties were made. The freezing point was -6.5°C, the slump was 180 mm, the air content was 10.4% (we lowered the AEA dose for this truck but the air content was still too high), the unit weight was 2280 kg/m³, and the mixture temperature was 11°C. The final concrete was placed at 1127.



Figure 57. Onsite disposal of excess concrete.

Because the second truck was expected to contain some concrete at the end of the job, and it was likely to stiffen inside the drum if it remained there too long, a place was prepared to discharge the waste concrete and wash water (Fig. 57).

The work of placing the concrete, not including the waiting time between the two trucks, took 31 minutes, between 1025 and 1127. Emplacement consisted of directing the concrete with the truck's chute, consolidating it with an internal vibrator (Fig. 58), and finishing the resulting surface with a magnesium float. No further work was done to the concrete. At approximately 1300, the finished concrete was covered with a sheet of plastic (to minimize evaporation) and with a 25-mm-thick insulation blanket. The insulation was not needed to protect the mass of the concrete against freezing, but to prevent freezing where steel bolts protruded from the finished surface. Air temperatures were expected to drop significantly below the freezing point of concrete. Unfortunately, we neglected to insulate the exposed ends of the steel form ties, which caused the concrete immediately next to them to cool below its freezing point.



Figure 58. Placing and consolidating the concrete.

### Strength Gain

To estimate strength development at various points in the concrete (Fig. 59), we tested samples for compressive strength at various ages from the group cast during placement. Seventeen cylinders were stored in picnic coolers next to the bridge, with two of them containing embedded thermocouples to monitor temperature history. Sets of

these field samples were periodically transported to the CRREL laboratory for testing; 26 cylinders were immediately returned to CRREL after the placement and stored in a 23°C room, with two of these cylinders similarly instrumented with thermocouples. All specimens remained in their plastic molds until testing. A maturity curve was developed from the strength data for samples stored in the picnic coolers, cylinders cured in the lab, and temperature data obtained from the dummy cylinders at both locations. Once the maturity curve was developed, the curbing's strength could be estimated for any thermocouple location in the bridge using its temperature-time history.

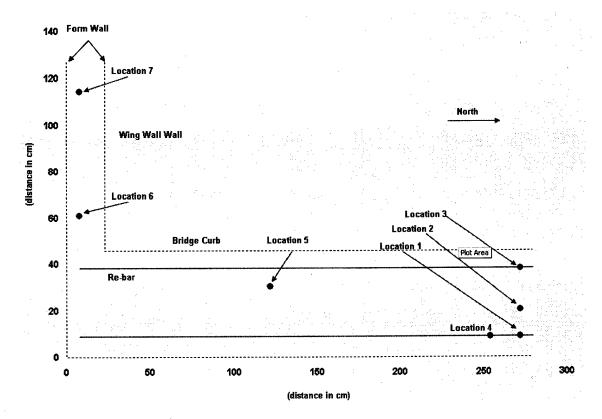


Figure 59. Temperatures were monitored in seven locations.

The resulting strength gain curves for three critical points in the curbing are shown in Figure 60. The middle and top surface of the curbing reached 20 MPa in less than 3 days. The coolest portion of the curb, that in contact with the existing concrete substrate, reached 20 MPa in approximately 5 days. The forms were removed from the concrete on the fifth day. Figures 61 and 62 show the temperatures of the outdoor air and three locations in the concrete.

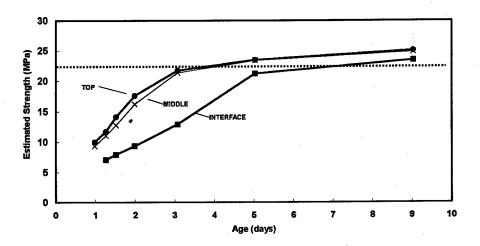


Figure 60. Strength gain curves for three critical points in the curbing.

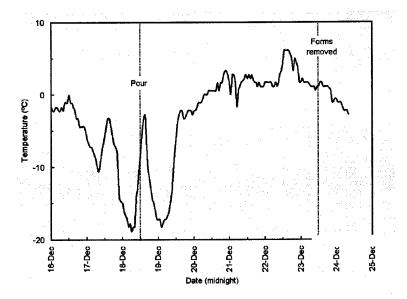


Figure 61. Ambient air temperatures (NOAA 2002b).

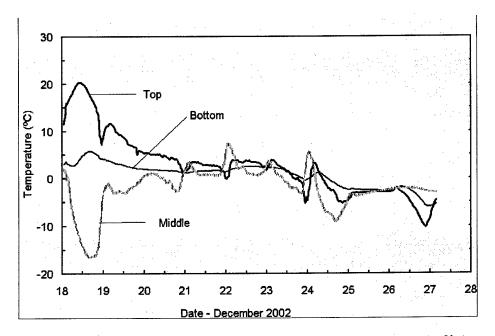


Figure 62. Temperatures from three levels within the curing concrete. Note that the middle temperature, which was attached to a steel form tie exposed to the outdoor air, dropped significantly below the freezing point of the concrete. The ends of the ties should have been insulated.

#### Lessons Learned

Zero-Delivery-Time Concrete. By not mixing the final admixture into the concrete until it reached the jobsite, we created a concrete that had full slump and thus a longer working time than if the mixing had taken place at the ready-mix plant. This technique presents the possibility of using less water—and thus fewer admixtures—to better control slump and reduce costs.

The Concrete Immediately Adjacent to the Form Ties Froze (Fig. 62). This is not a serious problem because the concrete there serves no structural purpose. It, however, is not acceptable, but fortunately can be easily avoided on future jobs. The freezing occurred because the ties were exposed to the cold outside air through the side of the forms. A simple insulation plug stuffed into these holes would solve this problem in the future

Minus Twenty Degrees and Rising. The concrete, except near the form ties, did not approach the freezing point. This suggests that the weather could have been colder. It also suggests that work should be directed toward defining how rapidly heat is evolved from the cement as it cures. This would allow one to ultimately predict thermal profiles and strength development at any location in a structure over time. The best rule-of-thumb that

we might derive from this test is that antifreeze concrete made to the specifications of this study can be safely placed on a frozen substrate if the air temperature is -20°C and rising.

Using this Mixture was a Substantial Savings in Time and Money Over Using the Conventional Winter Concreting Technique. The normal approach required 96 labor-hours to erect an enclosure around the curbing, followed by 36 labor-hours to remove it. Materials were extra. In addition, the enclosure was heated for 24 hours prior to placing the concrete and an additional 72 hours to partially cure the concrete. Using antifreeze concrete, less than 2 hours were required to place, finish, and cover the concrete. The antifreeze admixture was estimated to add about \$700 to the cost of the concrete. The concrete without the admixtures cost about \$720.00 plus approximately \$750.00 to heat.

Finishing Was Still an Issue; the Concrete Was Sticky When Worked with a Magnesium Trowel. One possibility is to wait a short time before trying to conduct finishing operations. Because the concrete sets so rapidly, the waiting period might only be 45 minutes to an hour.

Working Outdoors Rather Than Within a Heated Shelter Was Preferred. Comments from the workers suggested that working around the supports of a shelter while placing concrete is one of the most difficult aspects of winter concreting. Also, many felt that air quality inside confined shelters is less than desirable, as workers come down with an inordinate number of colds during the winter season. During the antifreeze jobs that were always conducted outdoors, no-one suffered for a headcold. For these reasons, NHDOT preferred working outside of the shelters. It was nice, however, to have a warming hut to take occasional breaks.

Using More Than One Truckload Was Not a Problem. Because the concrete tended to lose slump quite rapidly while in transit, the concern was that scheduling multiple truckloads might present a problem. However, the concrete remained plastic long enough to allow the second truck to deliver without problems. In addition, when the second admixture was not mixed into the concrete until the truck arrived at the jobsite, there was little chance that the concrete would become too stiff before placement began. Though there was some discussion about possible cold joints between the two placements, there was no evidence of that during several visual inspections up to the writing of this report.

The Second Truckload Produced Two Balls of Unmixed Concrete. We saw this same occurrence in Wisconsin. It was probably caused by the truck's mixing fins not being washed down after batching. This phenomenon is being investigated, though at this point it is not considered indicative of a large problem.

Air Contents Were Too High. This is not considered a problem. Using less airentraining admixture should solve this issue, especially given greater experience with the performance of a specific mixture at each new batch plant.

### Concord, New Hampshire

Up to this point, the initial planning stage and two cycles of laboratory and field work were complete in accordance with the original project proposal (Korhonen 2000). As anticipated, the effort resulted in antifreeze concrete formulations suitable for highway infrastructure application in cold weather. Our next step was to determine if this new antifreeze technology was sufficiently advanced to be immediately usable to DOT construction crews.

This technology transfer was assessed across the street from the New Hampshire Department of Transportation headquarters in Concord. NHDOT, in cooperation with the City of Concord, was building a 70-m-long precast concrete sidewalk containing multiple test sections. Commercially available products, designed to assist the visually impaired to navigate crosswalks more easily, were evaluated for their resistance to snow, ice, and repeated plowing. Our participation in this study was to instruct NHDOT in casting about 18 m of sidewalk with antifreeze concrete for entrance and exit ramps to the test section.

### The Sidewalk

The test area was located on state property along Hazen Drive at the intersection with Loudon Road. To prepare the ground to receive the new concrete, frozen material was removed and replaced with crushed aggregate. The section of sidewalk nearest Loudon Road (entrance ramp) was approximately 11 m long and the exit ramp at the end of the 70-m test section was approximately 7 m long (Fig. 63). The sidewalk measured 150 cm wide by 13 cm deep, and it contained a welded-wire mesh that was pulled halfway up into the concrete during placement.



Figure 63. Entrance ramp test section at ambient temperature of -20°C.

### The Mixture

The ready-mix plant was located 55 km away from the jobsite. Because we had experience with the same concrete mixture just a couple months earlier in North Woodstock, New Hampshire, no pretesting of the concrete was done. As it turned out, we probably should have done some additional testing, or at least have been more concerned about the aggregate moisture contents. To avoid too much slump loss during transit, no admixtures were placed in the concrete at the ready-mix plant. The only ingredients added were cement (392 kg/m³, Type II), sand, coarse aggregate, and water (25°C) to make 5.5 m³ of concrete with an expected w/c ratio of 0.25. The drum was briefly turned to mix the ingredients thoroughly but then halted for the trip to the jobsite. The warm water and the lack of drum agitation during transit were to prevent the concrete from freezing inside the truck, as the air temperature was around -20°C at batching time.

The truck arrived on the jobsite at 1020. Between 1035 and 1041, the first admixture was added into the drum by pail (Fig. 64) and the concrete was mixed for 3 minutes. At 1045, the mixture was backed up in the drum and a pail of plasticizer dumped onto the fresh concrete. The drum was turned for 2 minutes, backed up, and at 1048 the airentraining admixture, poured into a pail of sand, was added into the drum. Mixing continued until the final admixture was added between 1051 and 1059 by pump (Fig. 65). The drum was turned at mixing speed for another 3 minutes.

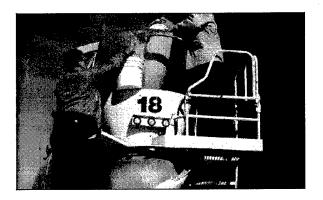


Figure 64. Adding the first admixture by pail.

Surprisingly, the concrete inside the truck was observed to have a slump of 25 mm. Based on our previous experience with this mixture, the slump should have been 100 to 150 mm. Additional plasticizer and a pail of water (19 L) were added. Because the mixture still looked stiff, two more pails of water were added into the drum. At 1112, the concrete placement began with the slump still much lower than desired (Fig. 66). After a few minutes, two more pails of water were added because the slump had dropped to less than 50 mm. Freezing point and strength gain samples were obtained at approximately

1114, and the concrete for the first section of sidewalk was placed by 1120. Figure 67 shows that the freezing point of this concrete was determined to be  $-6.5^{\circ}$ C. The w/c ratio was back-calculated to be approximately 0.34.

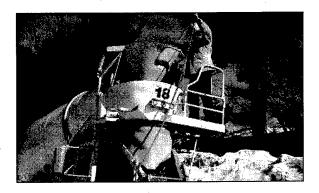


Figure 65. Pumping final admixture into truck.



Figure 66. The concrete was initially very stiff.

At that point, approximately 40 L of 65°C water were added to the remaining concrete (estimated to be 40% of the load), raising the concrete's slump to approximately 100 mm. Hot water from the truck was used because no more pails of cold water were available. By 1150, all remaining concrete had been placed in the exit ramp section and the waste (0.2 m³) was discharged into a temporary holding area (Fig. 68). Again, back-calculation suggested that the w/c ratio was 0.39 and the freezing point of the concrete was -5.6°C.

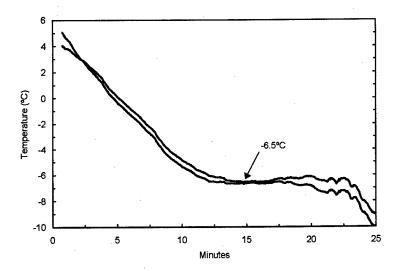


Figure 67. Initial freezing point for the entrance ramp concrete.

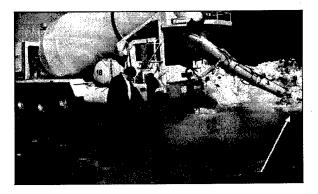


Figure 68. Cleaning mixer and discharging waste onsite.

# Finishing and Curing

Because the concrete was so stiff during the first half of the job, the concrete finishers had to apply extra effort to position and consolidate it in the forms. However, the concrete appeared to respond reasonably well to the vibrator. (Later, when the forms were removed from one portion, the vertical sides of the sidewalk appeared well-consolidated [Fig. 69]). The finishing consisted of using the truck's chute to place the concrete in its approximate final position, consolidating the concrete with an internal vibrator (Fig. 70), striking off excess concrete with a straightedge, and immediately using hand floats and

magnesium bull floats to smooth the surface (Fig. 71). Normally, the floating operation cannot be accomplished until all bleed water has left the concrete—a process that can delay finishing for several hours, depending on conditions. However, this concrete exhibited no bleeding, as had been the case with all of our other field trials, so finishing could be done as soon as practical. Following the floating process, the surface was given a broom finish, the slab was edged, and control joints were troweled into the surface (Fig. 72). The finishing ended around 1245, but the surface was still too soft to cover, so it was left alone for a while. At 1330 (approximately 2 ½ hours after the admixtures were mixed into the concrete), the sidewalk was covered with a sheet of plastic and a 25-mm-thick layer of insulation (Fig. 73). Very cold weather was expected the later that night.

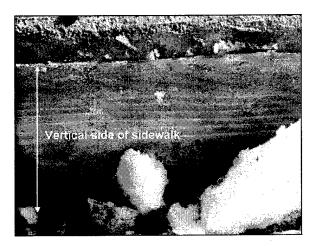


Figure 69. Sidewalk profile displays satisfactory consolidation, despite low slump.

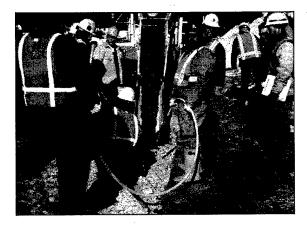


Figure 70. Preparing to consolidate the concrete with an internal vibrator.



Figure 71. Finishing the surface with magnesium hand floats and bull floats.

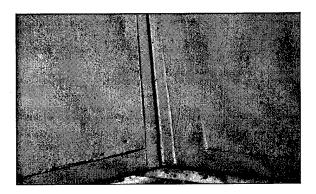


Figure 72. Finished sidewalk with broomed surface, troweled edges, and control joints.

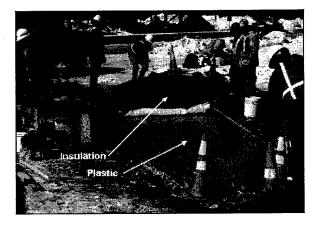


Figure 73. Sidewalk covered with a sheet of plastic and a layer of insulation.

# Strength Gain

To determine strength development, we tested samples for compressive strength at various ages from a group cast during placement. Combining these results with temperature data from dummy cylinders and from various points in the fresh concrete (Fig. 74), we developed a strength-maturity relationship for the mixture. Because the maturity method used to estimate in-place strengths has been shown to perform well whether the test cylinders are stored in conditions similar to the concrete structure of interest or in a laboratory at room temperature<sup>‡‡</sup>, we chose not to field-cure any cylinders. All test cylinders were stored indoors within standard laboratory curing rooms, both at CRREL in Hanover, New Hampshire, and NHDOT in Concord. One cylinder from each set of samples contained an embedded thermocouple from which a Nurse-Saul maturity function was developed, incorporating the strength data and temperature history of the test cylinders. This function was then used to estimate strength gain in the sidewalk.

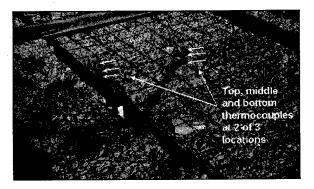


Figure 74. Thermocouples pre-positioned in the formwork.

The resulting strength gain curves for three points in the first section of sidewalk—representing the warmest, coldest, and an average location—are shown in Figure 75. The warmest location achieved 20 MPa in approximately 4 ½ days, and the coolest took about 7 ½ days to reach that same strength. Some of the forms were removed from the concrete on 19 February (the concrete was re-insulated), and the remaining forms and insulation were removed on 25 February, 10 days after concrete placement. Figures 76 and 77 show the temperatures of the outdoor air and the concrete at three locations, from warmest to coolest.

<sup>&</sup>lt;sup>‡‡</sup> For a detailed discussion, refer to Appendix D, which evaluates the ability of the maturity method to predict strength development at low temperatures.

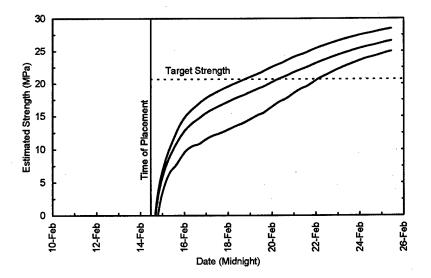


Figure 75. Strength development at three locations.

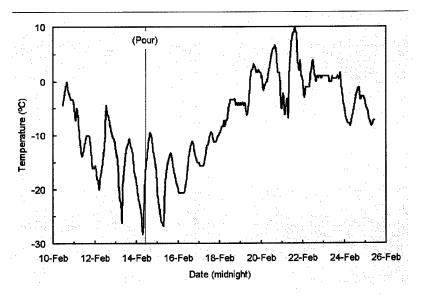


Figure 76. Ambient air temperatures at Concord, New Hampshire (NOAA 2003).

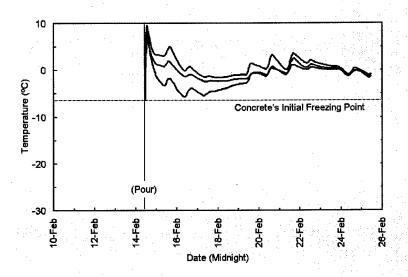


Figure 77. Concrete temperatures at three locations in the sidewalk.

# Project Control

The primary objective of this project, as previously stated, was to determine if this technology could be handed off to others for immediate field use. For this project, we developed a set of instructions for a Design Engineer in the Bureau of Bridge maintenance, NHDOT to follow. Figure 78 shows the information sheet, in the original English units, \$\frac{80}{2}\$ provided to the jobsite work crew. As we observed, things went according to plan. The concrete was ordered and delivered, and all admixtures were dosed into the truck in the proper amounts and sequence. Except for the mixture having a severely low slump (the cause is discussed below), we are convinced that others could use this technology.

#### Lessons Learned

Moisture Control. It is important to account for the total water content in the mixture when setting admixture dosages. Too much water could render the concrete unable to resist freezing, and too little water, though helpful toward achieving low freezing points, could make the concrete difficult to work. Water from the aggregate is the most difficult to control, particularly with snowfall in the 24 hours preceding a job. For example, in Rhinelander, Wisconsin, and North Woodstock, New Hampshire, we encountered concrete produced with too high a w/c ratio (determined through our freezing-point measurements). On the other hand, for this job the concrete was delivered with a much lower

<sup>§§</sup> Readers desiring SI values are urged to use the conversion table provided.

w/c ratio than expected. It appears that the moisture content of the coarse aggregate was inaccurately measured to be too high. Unfortunately, we could not confirm the actual w/c ratio of the concrete until after it was completely discharged from the truck.

Procedure for NHDOT					
Order 6 yd <sup>3</sup> of concrete with 658 lb cement per yd <sup>3</sup> , a w/c ratio of 0.25, and no admixtures.					
To this, NH DOT will add the following:					
1 yd <sup>3</sup> 6 yd <sup>3</sup>					
,		Nominal Dose	Volume (fl oz)	Weight (lb)	
	Air Entrainer	1 fl oz / yď <sup>3</sup>	6.0	0.41	
	Plasticizer	4.5 fl oz / cwt	177.7	14.68	
	Admixture #1	6 gal / yd <sup>3</sup>	4608.0	388.34	
	Admixture #2		3553.2	312.17	
Procedure for dosing admixtures is as follows:  1) Add admixture #1 into drum. Spin drum for 3 minutes.  2) Backup load to top of drum and pour plasticizer onto the concrete.					
Spin drum for 2 minutes.					
3) Pour air entrainer into bucket containing ~10lb of sand. Backup load again and add the sand / AEA mixture onto the concrete. Spin drum several revolutions.					
4) Add the admixture #2 into the drum. Spin drum for 3 minutes.					
Visually inspect the slump (it should be 4 to 6 inches).					
CRREL will make any additional adjustments if needed.					

Figure 78. Worksheet for producing antifreeze concrete.

Test Mixture. Because of variations in job conditions and materials, the performance of the concrete will vary from job to job. It is, therefore, important that each concrete mixture used for fieldwork be pre-tested in 2- to 3-m³ batches to check that the desired effects are being obtained. Conducting trials using job-specific materials will help determine the required admixture dosage for the desired freezing point, the proper dosage of air-entraining admixture, the fine-tuning of admixture combinations needed to achieve the desired slump, the length of time that the mixture will remain workable, and the time-temperature strength performance of the concrete. This will allow the job supervisor to know what to expect from the concrete when it is ordered, upon arrival at the jobsite, during placement and finishing operations, and throughout the curing process.

How Best to Order Concrete. Based on the difficulty we have encountered with achieving accurate moisture contents of aggregate in winter field situations, it is probably best that some water be withheld from the mixture at the ready-mix plant. Then, once all admixtures are dosed, either at the plant or at the jobsite, and provided that the mixture has been pre-tested, the job supervisor can either add water into the mixture until it comes up to the expected slump or do nothing if the slump is acceptable Most importantly, the concrete can be rejected if the slump is too high upon arrival, revealing an excess of free water.

Hot Water. Throughout this study we have advocated the use of cold mixing water to achieve a concrete mixture temperature between 5 and 10°C to avoid rapid stiffening. However, it is possible, as seen on the Concord job, to add hot water from the truck to the mixture, creating greater slump, without causing problems. This suggests that more study should be done to determine the benefit of using higher mixture temperatures. Perhaps hot water, especially when added at the jobsite, would allow fewer admixtures to be used but still achieve the same performance now produced by higher doses. Alternatively, hot water might allow lower curing temperatures, especially when used in tandem with full-strength antifreeze doses.

We Need a Way to Tailor the Admixture Job-by-Job. Of the five field projects conducted, only one job—the Concord sidewalk—came close to utilizing the full antifreeze potential of the admixture suite. In effect, that means that the concrete for the other four trials was over-designed and thus more expensive than it needed to be. Currently, we can't adjust the admixture dosage to account for the varying levels of protection that might be necessary for a given weather situation. We have a one-size-fits-all solution because the research necessary to forecast an internal concrete temperature as a function of outdoor air temperature has not been done yet. Users of this technology will need to be able to predict how a concrete mixture will perform in a particular environment—making it possible to optimize mixture design, economize material costs, and assure the desired outcome.

We Need to be Able to Measure the Freezing Point (and Thus the w/c Ratio) of the Concrete on the Truck. We have shown that it is possible to measure the freezing point of concrete in the field within about 10–20 minutes. It would be very useful to develop an instrument that would measure the freezing point of concrete automatically within 10 minutes. Having this information in such a timely fashion would allow one to determine the w/c ratio of the concrete and whether more water, and how much, could be added. Equally importantly, these data would also determine if the concrete should be rejected, in the case that more antifreeze cannot be added in sufficient quantity.

# 4 CONCLUSIONS

Five successful field tests demonstrated that it is possible to mix, transport, place, and finish antifreeze concrete at low temperature in full-scale operations using conventional materials, techniques, and equipment. The concerns in transferring this technology from the lab to the field were satisfied and the following lessons learned.

### **Full-Scale Batching**

Dosing admixtures onsite provided the best control and most flexibility with transport and placement; however, alternate methods proved feasible as well. Jobsite additions were trouble-free and required minimal time and effort with conventional admixture pumps. Avoiding excess water from snow and ice in the aggregate stocks was difficult, but could be accommodated by withholding a portion of the mixture water until last. Despite variations from the design water content, the concrete had adequate workability, no segregation, good early strength development, and acceptable freeze protection—never requiring outright rejection of a truckload.

# **Transporting**

Hauling distances of up to 55 km from the ready-mix plant were possible with some of the admixture dosing methods. Working times of over an hour were observed in some cases. Short transport times may allow the final dose of admixture to remain unmixed until arrival on the jobsite to counteract potential slump loss.



Figure 79. Finishing concrete immediately following placement.

There was no need to heat substrates or adjacent existing concrete before use, despite overnight temperatures of almost  $-30^{\circ}$ C prior to placement. However, any ice must be removed from forms and reinforcement prior to placing concrete. The concrete remained workable for 20 minutes and beyond during placing operations. Warm weather after placement is not harmful to antifreeze concrete. Antifreeze concrete did not exhibit bleeding, allowing finishing operations to be completed sooner than regular concrete under normal conditions (Fig. 79).

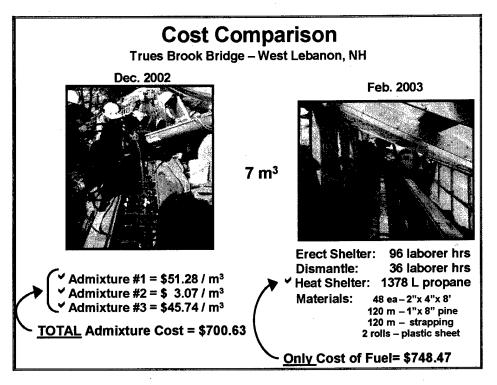


Figure 80. Cost comparison for the antifreeze concrete method versus the conventional approach. Note the admixture cost equaled the cost for heat in this instance, but in addition, tenting required significant labor and material to erect.

### **Cost Issues**

Using antifreeze admixtures provided significant cost savings versus conventional techniques. Whereas the customary tenting and heating approach can double the in-place cost of concrete per meter, using antifreeze technology instead cut this surcharge almost in half. In some cases, where the concrete is particularly exposed, shelters hard to erect, or the area to heat especially large, the savings can be substantial. Our clients estimated

for the Littleton, New Hampshire, repair that the heating approach would have cost 10 times more than the admixtures required.

The bridge curbing replacement in West Lebanon, New Hampshire, provided the opportunity to directly compare antifreeze method with a heated tent on identical pours (Fig. 80). The extra cost to place the antifreeze concrete was estimated at about \$110/m³ while the extra cost for the shelter alone (in materials and heat) was estimated at \$140/m³, besides the 132 labor-hours to erect and dismantle the shelter. It took only 1 hour to place 6 m³ of antifreeze concrete, but 3 hours to place the same quantity of normal concrete working in and around the tent structure (Fig. 81). Faster placement times means more productive (and ultimately less expensive) crews that are free to move onto other work sooner. Clearly, the antifreeze approach can make more jobs cost-feasible in winter that otherwise would not be attempted with conventional, cost-prohibitive heating methods.



Figure 81. NHDOT crew placing regular concrete (left) within the confines of a heated tent (right) during bridge curb repairs in West Lebanon. This pour, on the east side of the Trues Brook bridge, was the mirror opposite of the antifreeze concrete repair performed on the west side.

# **Compatibility with the Construction Process**

The use of antifreeze suites does not preclude the use of other low-temperature methods, such as insulation or heating, for further protection or performance. Additional methods could be employed after placement in cases where temperatures fall well below predicted values. An antifreeze concrete can be applied to most situations where standard concrete is used. Sequential batching of multiple truckloads was not a problem, making a continuous placement feasible.

# Cleanup

Cleanup of equipment in cold weather with standard methods was not a problem. Hot water from the concrete truck worked well and did not freezeup. Adding water to mixtures when rapid slump loss was encountered increased workability enough to allow full discharge and cleaning from the mixing drum.

# **Quality Assurance**

The use of the maturity concept is valid for estimating the strength of antifreeze concrete at material temperatures down to 0°C. Field-cured cylinders rarely represent the strength present in the corresponding structure without the use of maturity. Developing a maturity relationship with material taken at the time of actual placement is simple, timely, and more representative of the actual mixture used.

Freezing point measurements were a crucial quality control tool to test for variations in freezing point depression (and thus w/c ratio indirectly) from the desired value. Readings were possible at the jobsite in 10 to 20 minutes, though development of an automated device is needed before widespread use can be recommended. The ability to measure the freezing point quickly while the concrete is still in the truck would allow for adjustments or outright rejection *before* placement begins.

Overall, as is the case with all admixtures, we found that it was critically important to pre-test the suites in small field batches before project use to check that the desired effects were obtained. As with regular concrete, more field experience with a particular mixture should allow greater control of slump and entrained air. Keeping mixture temperatures low with cold water provided good performance, but higher values seem possible. Further study of adding hot water immediately before placement could allow the use of less admixture at lower cost or lower curing temperatures, or both. Regardless, the -5°C capability in antifreeze performance allowed operations to continue at ambient temperatures down to -25°C. This one size fits all approach works well, but further optimization of dosages based on temperature prediction models could result in better performance and lower costs for specific conditions and geometries. The demonstration of technology transfer in Concord, New Hampshire, showed that this new antifreeze technology is sufficiently advanced and user-friendly to be immediately usable by DOT construction crews.

### **Admixture Dosing Sequence**

Table 5 discusses the tradeoffs among three admixture dosing schemes. Plus, a fourth scheme was tried during the field project in West Lebanon, New Hampshire. Each scheme should be considered whenever any antifreeze concreting project is being planned. The primary consideration is to choose a scheme that produces a concrete that

allows time for the work crew to place, consolidate, float, and protect it before it stiffens. The approach that offered the most working time at the jobsite and was, thus, the easiest for the work crew to handle was the scheme that dosed all admixtures, including the airentrainer, into the truck at the jobsite.

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# **APPENDIX A: SUMMARY OF MATERIAL PROPERTIES**

Table A1. Portland cements used in laboratory and field tests.

Manufacturer	Lafarge North America	Ciment Québec, Inc. <sup>†</sup>	Cemex, Inc. "			
Source location	St. Constant, Quebec	St. Basile, Québec	Charlevoix, Michigan			
Type (ASTM C 150[2002a])	<b>-</b>	11	I ·			
Chemical e	compound (by ma	ass %)				
Silicon oxide (SiO <sub>2</sub> )	20.4	20.8	20.01			
Aluminum oxide (Al <sub>2</sub> O <sub>3</sub> )	4.8	4.3	4.53			
Ferric oxide (Fe <sub>2</sub> O <sub>3</sub> )	2.9	2.9	2.55			
Calcium oxide (CaO), total	62.6	62.7	62.83			
Magnesium oxide (MgO)	2.8	2.4	4.51			
Sulphur trioxide (SO <sub>3</sub> )	3.5	3.6	2.57			
Loss on ignition	1.1	0.7	0.99			
Insoluble residue	0.2	0.3	0.22			
Calcium oxide (CaO), free	0.7	1.1	N/A			
Alkali equivalent (Na <sub>2</sub> O)	0.86	0.8	1			
Potential c	ompounds (by m	ass %)				
Tricalcium aluminate (C₃A)	8	6	8			
Tetracalcium alumino-ferrite (C₄AF)	9	9.0	8			
Tricalcium silicate (C₃S)	54	53.8	62			
Dicalcium silicate (C <sub>2</sub> S)	18	18.9	11			
Physical characteristics						
Specific surface area, m²/kg	370‡	345***	383‡			
Specific gravity	3.15	3.15	3.15			

<sup>\*</sup> Used throughout laboratory testing and in West Lebanon, N.H., field test.

<sup>†</sup> Used in Littleton, North Woodstock, and Concord, N.H., field tests.

<sup>\*\*</sup> Used in Rhinelander, Wis., field test.

<sup>‡</sup> Blaine method.

<sup>\*\*\*</sup> Air permeability method

Table A2. Aggregates used in the laboratory testing phase.

	Fine Ag	gregate	Coarse /	Aggregate		
Source		ned Stone West on, NH	Lebanon Crushed Stone Wes Lebanon, NH			
Description	Natura	al sand	19 mm crushed ledge stone			
Bulk specific gravity (ssd)		2.72	2.97			
% Absorption		0.9		0.7		
Dry-rodded density (kg/m³)	1735		1680			
	Siev	e Analysis as per	ASTM C 136 (200	)1c)		
Sieve size	% Retained	% Passing	% Retained	% Passing		
19.0 mm		`	0.0	100.0		
12.5 mm			30.2	69.8		
9.5 mm		·	32.5	37.3		
4.75 mm	0.0	100.0	34.1	3.2		
2.36 mm	11.0	89.0				
1.18 mm	22.7	66.3				
600 µm	24.8	41.5				
300 µm	22.9	8.6				
150 µm	12.1	6.5				
75 µm	3.2	3.3				

# APPENDIX B: LABORATORY PROCEDURE FOR MEASUREMENT OVER TIME OF SLUMP, VOLUMETRIC AIR CONTENT, AND INITIAL FREEZING POINT

### I. Pre-Mixing

### A. Measuring Bulk Ingredients

Before weighing out aggregates, make sure moisture content is uniform, obtain a representative sample, and determine the amount of free water present. Measure the proper mass of coarse and fine aggregates into sealed containers to keep them at the measured moisture content until use. Similarly, measure out the cement by mass into containers and seal to keep it dry. Determine the amount of water needed and measure the proper mass out into containers and seal. Reserve approximately 500 g of this mixture water to use for rinsing admixture beakers during the batching process. Label buckets appropriately with information regarding ingredient, mass, moisture content, batch number, etc.

Allow all materials to equilibrate to the temperature of the laboratory overnight, if necessary. Cold and hot water may be mixed to obtain room temperature water for immediate use.

## B. Measuring Admixtures

### 1. Plant Admixtures

Admixtures dosed to simulate being added at the ready-mix batching plant are termed "plant" admixtures.

Plant admixtures dosed in relatively small volumes are measured into beakers prewetted with water so they can be fully rinsed with water when dosed into the mixture, ensuring all the admixture makes it into concrete. These typically include:

- Air entraining agent (AEA).
- Mid range water-reducing (MRWR).
- High range water-reducing (HRWR).
- Shrinkage-reducing.
- Retarding.

Plant admixtures dosed in relatively large volumes are measured into containers prewetted with the admixture itself so they can be directly added to mixture without a water rinse. These typically include:

- Corrosion inhibitor.
- Accelerator.

#### 2. Jobsite Admixtures

Admixtures dosed to simulate being added upon arrival of the concrete mixing truck on the jobsite are termed "site" admixtures. AEA added "at the site" is measured out onto 100 mL of dry sand (for a 0.042 m<sup>3</sup> mixture volume) in a beaker, shortly before dosing into the drum.

Site admixtures dosed in relatively small volumes are measured into beakers prewetted with admixture so they can be accurately dispensed into the mixture without any rinse water. (Additional water at this point would change the w/c ratio of the final concrete.) These typically include, but are not limited to:

- Mid range water-reducing (MRWR).
- High range water-reducing (HRWR).

Site admixtures dosed in relatively large volumes are measured into containers prewetted with admixture so they can be directly added to mixture without rinsing, such as:

- Corrosion inhibitor.
- Accelerator.

# C. Buttering the Mixer

The drum of the rotary mixer should be "buttered" to compensate for the loss of mortar from the test batch of concrete. Our buttering mixture consisted of a small batch of mortar mixed in the drum with the following proportions: 9 kg sand (ssd), 2 kg cement used in concrete mixture, and water slowly added "by eye" until a good workable mortar consistency was achieved.

### **II. Concrete Mixing and Testing Procedure**

The following procedure (Table B1) was used to perform the combined workability, entrained air content, and initial freezing point screening test described in the Selecting Effective Combinations (Laboratory I) section. Compressive strength samples, prisms for freeze—thaw durability, and critical maturity cylinders were made with concrete mixed generally following these procedures, up to the point where fully mixed antifreeze con-

crete resulted\*\*\*. As much as possible, the mixer was kept covered during rest periods—when material for samples wasn't being removed or returned to the drum. Jobsite doses of admixtures were adjusted to account for loss of mixture volume due to sampling and testing (i.e., the admixtures were dosed based on the estimated amount of cement or volume of concrete remaining in the mixer). Take care that each admixture is dosed separately into the concrete, avoiding direct contact with other admixtures.

Table B1. Timing for combined slump, air, and freezing point screening test.

	Task/comment	Mix	Test (and type)	Running time (in minutes)
	Wet down mixer drum with water and allow to drain.			
g)	Add buttering mixture sand.			
erin	Mix sand alone for 1 minute.	Х		
er butt	Stop mixer and add buttering mixture cement.			
mixe	Mix sand and cement dry for 1 minute.	X		
Preliminary (mixer buttering)	Gradually add water while mixing until mortar consistency is acceptable.	x		
relimi	Stop mixer and scrape any dry or unmixed material from sides of drum.			
۵	Mix for a final 2 minutes, ensuring mixer has an even coating. Dump excess mortar from drum.	x		
("	Add AEA to concrete sand, rinse beaker with water and add rinse water to sand.			
"plant	Charge drum with fine and coarse aggregates.			·
sit (	Mix aggregates dry for 1 minute.	X		−2 to −1
n tran	Add ½ of concrete mixture water to drum while continuing mixing.	х		-1 to -1∕₄
ite i	Stop mixer briefly and add cement to drum.			-1/4
Batch plant concrete in transit ("plant")	Start mixer (Zero is assumed to be the time that water and cement come together).	x		0 START TIME
plant	Add remaining ½ of concrete mixture water to drum.	x		0 to 1⁄4
Batch	Continue mixing. Add plant admixtures in the following sequence:	x		1⁄4 to 3
	Mid-range WR (rinse with reserved water).			

See the Confirming Low Temperature Performance (Laboratory II) section for any deviations in mixing procedure for a particular test.

	Task/comment	Mix		est (and type)	Running time (in minutes)
	High-range WR (rinse with reserved water).				
	Other admixtures (shrinkage reducer,	e reducer,			
	retarding, etc.).				
	Any remaining reserved mixture water.			:	
	Corrosion inhibitor or accelerator (no rinse).			<u></u>	
	Stop mixer and allow concrete to "rest" 3 minutes.		:		3
	Scrape any dry or unmixed material from drum sides (turn mixer ½ turn if necessary for access).				3 to 6
	Start mixer. Finish mixing concrete for 2 minutes.	x			6
•	Stop mixer. Begin filling and rodding slump cone and air meter samples.				8
"plant")	Pull first plant slump. Measure, record, and return material to mixer. (This is considered to be 0 minutes of "transit" time).		Х	Slump	10
sit ('	Continue preparation of air content sample.				
Batch plant concrete in transit ("plant")	Run mixer for 45 seconds (this is performed every 5 minutes, to simulate on average ~ 4 RPM of a truck during delivery and to keep concrete workable).	х			12 to 12 ¾
concr	Finish preparing air sample. Shake and roll air meter.		Х	Air	~ 15
ant	Run mixer 45 seconds.	Х			17 to 17 ¾
Batch pl	Prepare plant freezing point samples, place in coldroom.	-	Х	Freez- ing point	~ 20
	Run mixer 45 seconds.	Х			22 to 22 ¾
	Prepare slump cone sample.		V		23 to 25
	Pull second slump. Measure, record, and return material to mixer.		Х	Siump	25
'	Run mixer 45 seconds.	Х			27 to 27 ¾
	Run mixer 45 seconds.	Х			32 to 32 ¾
	Prepare slump cone sample.				33 to 35
	Pull third slump. Measure, record, and return material to mixer.		х	Slump	35
	Run mixer 45 seconds.	Х	***************************************		37 to 37 ¾
	Run mixer 45 seconds.	X	***************************************		42 to 42 ¾
	Prepare slump cone sample.		***************************************		43 to 45

	Task/comment	Mix		est (and type)	Running time (in minutes)
	Pull fourth slump. Measure, record, and return material to mixer.		Х	Slump	45
	Run mixer 45 seconds.	Х			47 to 47 ¾
*,	Record entrained air content of concrete. Empty and clean air meter to accept sample following further admixture doses.		Х	Air	~ 50
"Plant"	Run mixer 45 seconds.	X			52 to 52 ¾
Ē.	Prepare slump cone sample.				53 to 55
	Pull fifth and final plant slump. Measure, record, and return material to mixer.		Х	Slump	- 55
	Start mixer. Add site admixtures in sequence:	x			55 ½ to 57
	AEA on sand in beaker.				
	Mid-range WR (no rinse).				
	High-range WR (no rinse).				
	Accelerator or corrosion inhibitor (no rinse).				
	Continue mixing to ensure concrete is fully mixed.	x	****		57 to 59
	Prepare slump cone and air meter samples.				59 to 61
fe")	Pull first site slump. Measure, record, and return material to mixer.		Х	Slump	61 .
s,	Continue preparation of air content sample.				
ete	Run mixer 45 seconds.	Х			63 to 63 ¾
Jobsite concrete ("site")	Finish preparing air sample. Shake and roll air meter.		X	Air	~ 65
site	Run mixer 45 seconds.	Х			68 to 68 ¾
형	Prepare slump cone sample.				69 to 71
	Pull second site slump. Measure, record, and return material to mixer.		Х	Slump	71
	Run mixer 45 seconds.	Х			73 to 73 ¾
	Prepare site freezing point samples, place in coldroom.		Х	Freez- ing point	~ 75
	Run mixer 45 seconds.		***************************************		78 to 78 ¾
	Prepare slump cone sample.				79 to 81
	Pull third site slump. Measure, record, and return material to mixer.		Х	Slump	81
	Run mixer 45 seconds.	Х			83 to 83 ¾

	Task/comment	Mix		est (and type)	Running time (in minutes)
	Run mixer 45 seconds	Х			88 to 88 ¾
	Prepare slump cone sample.				89 to 91
	Pull fourth site slump. Measure, record, and return material to mixer.		Х	Slump	91
	Run mixer 45 seconds	Х			93 to 93 ¾
	Run mixer 45 seconds	Х			98 to 98 ¾
	Run mixer 45 seconds	Х			103 to 103 ¾
te")	Prepare slump cone sample.				104 to 106
Jobsite concrete ("site")	Pull fifth site slump. Measure, record, and return material to mixer.		х	Slump	106
E	Run mixer 45 seconds	Х			108 to 108 ¾
8	Record entrained air content of concrete.		Х	Air	~ 110
site	Run mixer 45 seconds	Х			113 to 113 ¾
형	Run mixer 45 seconds	X			118 to 118 ¾
	Prepare slump cone sample.				119 to 121
	Pull sixth and final site slump. Measure and record.		Х	Slump	121

# APPENDIX C: DETAILED PROCEDURES FOR FREEZING POINT DETERMINATION OF CONCRETE IN THE LABORATORY AND FIELD

#### Introduction

Developing a method to swiftly measure the initial freezing point of a concrete mixture in the field was presented as an "optional" element in our original project proposal (Korhonen 2000). In the laboratory, this test served as confirmation that the concrete mixture would be protected down to a temperature of -5°C. However, after using this measurement during our preliminary fieldwork and the five field trials previously described, it quickly became clear that the freezing point measurement was an invaluable quality control tool for verifying that a particular concrete mixture was properly batched and dosed.

The laboratory method was modified into a practical field method, performed concurrently with other standard quality control tests, such as slump measurement or entrained air content. The primary objectives of the field freezing point test are to:

- Ensure that the performance of the site-delivered concrete mixture meets the specified design freezing point, a minimum of -5°C for this project.
- Accurately measure the freezing point of the concrete mixture within a tolerance range of  $\pm 0.3$  °C.
- Provide a relatively simple test procedure which yields results in approximately 10–15 minutes.
- Provide an indirect method for determining the w/c ratio of a mixture if the admixture doses and batching weights are accurately measured.
- Permit adequate time to adjust the levels of either admixtures or water in the mixture prior to releasing it into the forms.

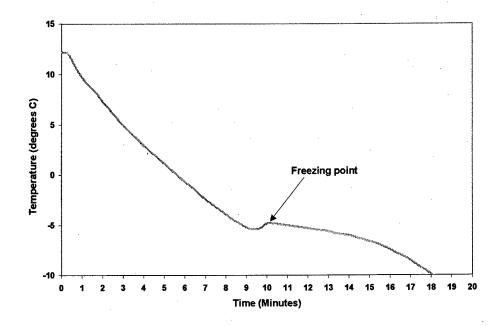


Figure C1. A typical freezing point measurement curve for a sample of -5°C antifreeze concrete.

## **Cooling Rates**

In the lab, a large coldroom is used to freeze the test cylinders. This was previously discussed in *Selecting Effective Combinations (Laboratory I)*, *Initial Freezing Point* and illustrated in Figure 7. In the field, dry ice is used to quickly freeze the test cylinders (Fig. C1). In either case, three cylinders are tested. Figure C2 illustrates the difference in the cooling rates between the laboratory and the field. Generally in the field, the field mixture is initially colder, around 10°C, and the overall cooling temperature range is smaller. Dry ice was selected because it creates a portable cooling environment, it is economical, and readily available. Temperatures within the cooler will not be uniform; the bottom closest to the ice will be colder than the space above. This will affect how quickly the cylinders freeze and the clarity of the freezing point reading.

In the lab, test cylinders typically cool at a rate of  $-0.5^{\circ}$ C per minute. In the field, the sample must cool faster, at rates up to  $-2^{\circ}$ C per minute to obtain a freezing point reading in about 10 minutes. While rates colder than  $-2^{\circ}$ C per minute have been tried in the field, the freezing point temperature is difficult to clearly determine.

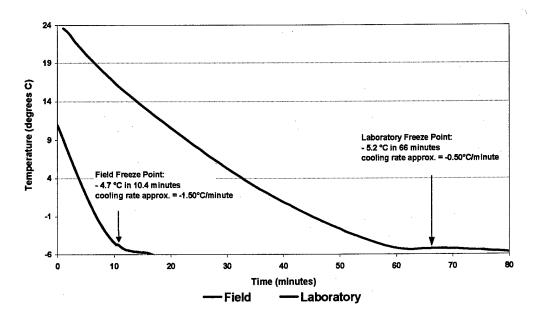


Figure C2. Examples of antifreeze concrete freezing point curves from the laboratory and the field illustrating the different cooling rates.

## **Temperature Measurement Devices**

The equipment used to measure the freezing point consisted of a thermocouple and a datalogger to record temperatures, and a computer for data processing. In general, the data collection system should meet the following requirements: be easily transportable into the field, operate over a wide temperature range, provide sufficient storage capacity, have good accuracy and resolution, and be programmable to accommodate various sampling rates. As an example of different sampling rates, 1-minute readings were found to be satisfactory for the lab, while 1-second readings were needed in the field. Three types of dataloggers were assessed during this study: ACR Systems, Inc. SmartReader<sup>TM</sup> Plus 6 Thermocouple Logger (ACR), Omnidata® International, Inc. EasyLogger<sup>TM</sup> 900 Series (Omni), and the Campbell Scientific, Inc. CR10 (CSI).

Table C1 lists the specifications for each of the three dataloggers. All three systems met the criteria of transportability, operating temperature range, and storage capacity. For programming, viewing, and downloading data, each system came with its own proprietary software. Both the Omni and CSI systems were capable of sampling at a rate of 1 second, while the fastest sampling rate with the ACR was 8 seconds. Accuracy is the difference between the instrument reading and the actual value. Good accuracy is best achieved by operating the device within the appropriate temperature range and environmental conditions. The resolution is the smallest quantity that the device is capable of measuring. The differences between accuracy and resolution, and sampling rates for the

systems became more apparent when the loggers were used during the lab study to develop a feasible field procedure. As reported in the manufacturer's specifications, the accuracy and resolution for each system is listed in Table C1 along with the error.

Table C1. Datalogger specifications used in laboratory investigation.

	ACR Systems, Inc. SmartReader™ Plus 6ª	Omnidata International Inc. EasyLogger™ 900 Series <sup>c</sup>	Campbell Scientific, Inc. CR10 <sup>e</sup>	Omega Engineering, Inc. Thermocouple Wire <sup>h</sup>
Operating temperature range	–40 to 70°C	–25 to 50°C	–25 to 50°C	–270 to 400°C
Internal data storage	128K	104K RAM	64K RAM	_
External data storage	N/A	256K	716 MB <sup>f</sup>	<u></u>
Differential channels	7	6	6	
Power source	3.6-V Internal lithium battery	12-V (D-cell) batteries	External 12-V power	
Reference temperature	Internal	Internal	External	_
Resolution	±0.40°C	±0.06°C	±0.03°C	
Accuracy	±3.0°C <sup>a</sup>	±0.5°C	±0.4°C	±0.01°C
Error (°C)	±3.40°C	±0.61°C	±0.51°C	±0.01°C
Sampling rate (seconds)	8	1	1	
Software	TrendReader® b	EasyTools LT <sup>d</sup>	PC208W <sup>g</sup>	

a (ACR 1999a)

Sources of error from these systems come from the datalogger, the thermistor reference junction, and the thermocouple. For the ACR, the error is the accuracy of the full scale operating range of the thermocouple plus the resolution, or  $\pm 3.4^{\circ}$ C. The CSI and Omni systems have errors of  $\pm 0.51$  and  $\pm 0.61^{\circ}$ C, respectively. This consists of a logger error of  $\pm 0.1\%$  of the full scale input range, or  $\pm 0.4^{\circ}$ C for the CSI and  $\pm 0.5^{\circ}$ C for the Omni; plus  $\pm 0.1^{\circ}$ C for the accuracy of the reference thermistor (the CSI system used a CSI 10TCRT); plus  $\pm 0.01^{\circ}$ C accuracy of the thermocouple (Stallman and Itagaki 1976). These error ranges are higher than our target of  $\pm 0.3^{\circ}$ C. The reference temperature also carries a certain amount of error into the temperature reading. This error is not always included in the published specifications for the device. No published information on the Omni thermistor is available.

b (ACR 1999b)

c (Morgan 1992)

d (Wescor 2001)

e (Campbell 1997)

f (Campbell 1991)

g (Campbell 2001)

h (Omega 2001)

i (Stallman and Itagaki 1976)

Thermocouples are composed of two different metals that are joined to create a path for an electromagnetic force to flow. There are two junctions, one at the tip where the metals are in contact with each other and the other where the wires connect to the datalogger. A change in temperature between the two junctions creates a change in the voltage output (usually in millivolts). When the temperature remains constant there is no change in the voltage. A reference temperature is required to detect the change in temperature. Most commercially available dataloggers are programmable with internal functions to automatically convert the voltage output into a temperature. This is useful for easily viewing and analyzing the output data.

Temperature measurements for both the laboratory and field freezing point readings used 24-gage, high quality, type T, copper-constantan thermocouple wire with polyvinyl insulation available from Omega Engineering, Inc†††. Advantages to using thermocouples are that they are economical, easy to fabricate, rugged, and operate over a wide temperature range. High quality thermocouple wire offers an accuracy of  $\pm 0.01^{\circ}$ C (Stallman and Itagaki 1976), as noted in Table C1. For this project, the temperature range is well within the capabilities of thermocouples. A disadvantage is that thermocouples are susceptible to noise. To make a simple thermocouple, strip the insulation off of one end of the wire, twist the wires tightly together with a pair of pliers and clip the end to create good contact between the metals. The leads of the other ends are stripped and wired to the datalogger.

Thermocouples require a reference temperature as a comparison for temperature measurement. All three of the logger systems used thermistors as the reference junction. The reference temperature may either be external, where it is manually wired to the datalogger (CSI) or internal (Omni and ACR). For best results, the reference temperature should remain stable. External reference temperatures tend to be more susceptible to rapid temperature changes, but this is resolved by isolating them.

Thermistors were also considered for measuring the freezing point in a sample. Unlike a thermocouple, a thermistor does not require a reference temperature. However, thermistors are, in general more expensive, more fragile, and not reusable once the sensor is cast in concrete. During the lab study, a Campbell Scientific 107 thermistor probe was tested, but the thermistor sensor was protected by an epoxy coating that slowed the response time of the reading. As a result of this, commercially available thermistors may need to be modified before using for the freezing point tests.

The differences in sampling speed and resolution are illustrated in Figure C3. Here, a freezing point test was conducted using the ACR and Omni dataloggers both set to sample at their fastest rate: 8-seconds for the ACR and 1-second for the Omni. The figure

<sup>&</sup>lt;sup>†††</sup> Refer to the notes at the bottom of Table C1 citing product literature about wire, logger, and software systems discussed here.

shows that 1-second sampling rates and finer resolution from the Omni datalogger provide more detail in the curve in order to determine the freezing point.

Because the ACR was found not suitable for freezing point readings, additional freezing point tests were conducted using both the Omni and the CSI dataloggers. While the Omni datalogger was capable of 1-second readings, the output was inconsistent when several channels were used simultaneously. For this reason, the CSI system was selected for freezing point measurements. The system is composed of a CR10 datalogger connected to a 12-V power supply and an SM 192/716 (716MB) storage module. It was programmed to read five differential channels.

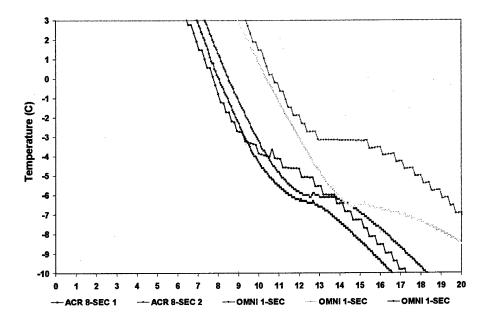


Figure C3. Laboratory results comparing the sampling speed and resolution of the ACR and Omni dataloggers. This test used test tubes set into 51- x 102-mm cylinder sleeves.

# Sample Size

In the lab, three 76-  $\times$  152-mm cylinders are placed on wire shelves in the coldroom and they cool in air. A field setup was tested in the lab using different sample sizes, ranging from 25.4-  $\times$  89-mm round-bottom tubes (Nalgene Centrifuge Ware) up to larger 76-  $\times$  152-mm plastic cylinders. The tubes have a small opening that only paste will fit through. Sieving the mixture for paste takes more time and is difficult when the mixture is stiff. Therefore, the tubes are not recommended for field use. Cylinders 76  $\times$  152 mm and larger required more than 20 minutes to obtain a freezing point reading and were

eliminated from further testing. In the field,  $51-\times 102$ -mm cylinders were successful because they are easy to handle when using either mortar or concrete.

For either lab or field measurements, the location of the thermocouple tip should be in the center of the cylinder and be surrounded by as much paste as possible. If the tip is located against a piece of stone in the mixture, this may affect the freezing point reading because the thermal properties of larger aggregate are slightly different from the paste. To maximize the contact with the paste in the mixture create a 'cage' by coiling the thermocouple wire about five times (the diameter should be about the size of your index finger) and tuck the thermocouple tip up into the center of the coil. Fill the cylinder roughly half full with either concrete or paste, and lightly tamp about six times to remove any air bubbles. Insert the 'cage' in the middle of the sample and fill the remainder of the cylinder. Lightly tamp about six times to remove any air bubbles and cap the sample. Feed the thermocouple wire through a hole poked in the center of the cap (set this up prior to the test), as the wire is less likely to get pinched when the cap is secured. Place the cylinders in a cold environment and monitor the temperature for the freezing point.

In the lab, the cylinders are placed on wire shelves in a  $-20^{\circ}$ C coldroom with adequate space between them to permit air flow, and temperature readings are taken every minute. In the field, a cooler of dry ice serves as a portable cooling environment. Initially, the cylinders were placed in direct contact and completely covered with the dry ice to cool them (Fig. C4). This approach yielded cooling curves with cooling rates faster than  $2^{\circ}$ C per minute. As the freezing point is a change in temperature of as little as 1 or 2 tenths of a degree, and may hold this temperature for less than 30 seconds, curves may be similar to those obtained when a  $51-\times 102$ -mm cylinder is placed directly in dry ice (Fig. C4), such that it is difficult to determine the freezing point. While the curve of the tube placed directly in dry ice in Figure C4 provides a good freezing point reading, this was not repeatable with the small sample sizes.

To slow the rate of cooling, the samples were placed into sleeves to act as a buffer (Fig. C4). The test tubes were placed into 51- × 102-mm sleeves and the 51- × 102-mm cylinders were placed into 76- × 152-mm sleeves. This increased the cooling time needed for the 51- × 102-mm test cylinders to a little over 30 minutes. In the field, the buffer was created by decreasing the amount of dry ice in the cooler and elevating the cylinders when their temperature approached 0°C. A thin layer of dry ice was spread on the bottom of the cooler and allowed to set for 15 to 20 minutes before the samples were cast. When placing the cylinders in the cooler, the ice is moved aside and the cylinders set vertically on the bottom of the cooler, but not in direct contact with the cylinders, for about 10 minutes. Temperatures are monitored with a laptop computer. As the temperature of the cylinder approaches 0°C, the cylinders are then elevated (onto a 50-mm piece of foam) to slow down the cooling rate and clarify the reading. While this aids in obtaining a clear freezing point reading, it also disturbs the samples when the cooler lid is opened to

rearrange the cylinders. Once the temperatures have reached well beyond the expected freezing point, the data are downloaded and processed to determine the freezing point measurement.

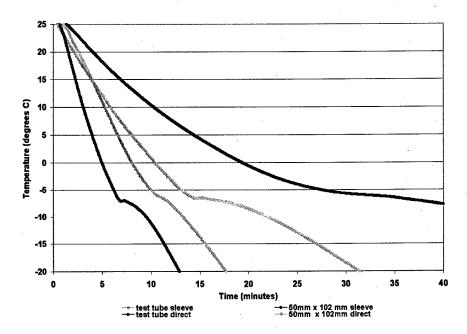


Figure C4. Lab results showing the freezing point readings of different sizes of cylinders and orientation in the dry ice.

### **Conclusions**

The development of the procedure described above offers a technique and some considerations to measure the freezing point either in the lab or in the field. To obtain an accurate freezing point measurement, it is important to understand the rate of cooling of the sample, the full capabilities of the type of equipment used for the measurement, and the size and orientation of the sample in the cooling environment. Currently, the methods employ sensitive electronic equipment requiring special skills to program and operate. A single, simple, rugged device is needed that, once a small sample of the mixture is placed inside, automatically generates a reading in 10–15 minutes without user-processing of the data. An accurate device such as this would considerably simplify freezing point measurement, making a valuable tool available for use throughout the industry.

# APPENDIX D: MATURITY METHOD FOR LOW-TEMPERATURE CONCRETE

### Introduction

Following the placement and finishing of the concrete, a method was needed to determine the in-place strength of the concrete so that, as with normal concrete, forms may be removed and construction operations may continue. The maturity method is a widely accepted, non-destructive way to estimate the in-place strength of freshly placed concrete based on the curing temperature and concrete age. Current practice discusses the use of the maturity method in determining removal of cold weather protection (ASTM C 1074 [1998b], ACI 306 [1988]).

An initial lab study was conducted that applied the maturity procedures in ASTM C 1074 (1998b) to antifreeze concrete, with some modifications. Based on this study, a procedure was developed to use the maturity method in the field.

# **Maturity Methods**

The maturity method is based on the relationship that exists among curing time, concrete curing temperature, and the rate of strength gain. The curing time and concrete temperature determine the rate of cement hydration, which then determines the rate of strength gain. Normal concrete cured at lower temperatures requires more time to gain appreciable strength as compared to concrete cured at higher temperatures. Our antifreeze concrete formulations have shown appreciable strength gain at low temperatures in a matter of days. Two acceptable maturity methods for estimating the in-place strength are the *time-temperature factor* and the *equivalent age*. These methods are similar as they both use the age (curing time) and concrete temperature as inputs to estimate strength. Both of these methods were evaluated for their potential use with antifreeze concrete.

One advantage to using the maturity method is the ability to monitor multiple locations in a structure, in particular, critical locations most susceptible to cold weather damage. Both maturity methods begin by developing strength-maturity relationship curves under controlled conditions in the lab. Either maturity method is used with a thermal history to calculate a *maturity index*. The primary assumption of the maturity method is that similar samples of concrete will obtain the same level of strength at the same maturity index (ASTM C 1074 [1998b], Malhotra and Carino 1990). Maturity indexes may be different for different mixtures.

The time-temperature factor (eq D1) is a simple relationship that assumes the strength gain of concrete is linear. The input values for the equation are the average temperature  $(T_a)$  over a time period  $(\Delta t)$  and a datum temperature  $(T_0)$ . The datum tempera-

ture is the lowest temperature where strength continues to increase with time, below which there is no increase in strength with time (Mindess and Young 1981). ASTM C 1074 (1998b) provides a method to determine the datum temperature in the laboratory. Datum temperatures of -10°C (Mindess and Young 1981) and -5°C (ACI 306 [1988]) have been recommended for use with normal concrete. As the antifreeze concrete mixtures under investigation in this study contain higher dosage levels of admixtures designed to cure at lower temperatures, the datum temperature was determined in the laboratory and compared with these accepted values.

$$M(t) = \sum (T_{\rm a} - T_{\rm 0}) \Delta t \tag{D1}$$

where:

M(t) = cumulative time-temperature factor at a specific age t (maturity index) (degree-hours)

 $\Delta t$  = time interval (hours)

 $T_a$  = average temperature of concrete during the time interval ( $\Delta t$ ) (°C)

 $T_0$  = datum temperature (°C).

There are advantages and disadvantages to using the time-temperature factor. Among the advantages, it is a fairly simple function to calculate and should an incorrect datum temperature be used, there is a correction method (Penn State 2003). Disadvantages are that the function provides a good approximation when used in a temperature range between -5 and 30°C, and as it is a linear function, errors are possible when there is either a steep slope change in the temperature or lots of fluctuation (Penn State 2003).

The equivalent age function (eq D2) is exponential and uses the activation energy. Equivalent age is defined as, "the amount of time needed for a given sample of concrete at a specific temperature to attain an equivalent level of maturity that a tested sample has achieved" (Penn State 2003).

$$t_{\rm e} = \sum e^{-Q\left(\frac{1}{T_{\rm a}} - \frac{1}{T_{\rm s}}\right)} \Delta t \tag{D2}$$

where:

 $t_{\rm e}$  = equivalent age at a specific temperature  $T_{\rm s}$  (hours)

Q = activation energy (K)

 $T_a$  = average temperature of concrete during the time interval ( $\Delta t$ ) (K)

 $T_{\rm s}$  = specified temperature (K)

 $\Delta t$  = time interval (hours)

Because the equivalent age function is an exponential, an advantage is that it gives a better approximation over a larger temperature range, as the activation energy accounts for the amount of energy required for hydration, and this gives more meaning to the equivalent age value (Penn State 2003). Among the disadvantages of the method, it is a more complex equation, which may lead to confusion; and no correction is available should an incorrect activation energy be used. However, the activation energy may be calculated, as illustrated in ASTM C 1074 (1998b). Suggested values for Q are 4700 K (Con-Cure Corporation) and 5000 K (ASTM C 1074 [1998b]); and  $T_s$  are 20 or 23°C (ASTM C 1074 [1998b], Penn State 2003).

# **Laboratory Study**

The initial lab study used an antifreeze concrete mixture, with proportions similar to the WRG III suite, and followed ASTM C 1074 (1998b). Three sets of  $76-\times152$ -mm compression strength cylinders were cast. All cured in air, one set cured at 23°C, and because these mixtures are intended for low-temperature use, the other two sets of cylinders cured in a coldroom held at -5°C and in an insulated box held at 5°C (located within the -5°C cold room). A curing temperature of 5°C was selected as this is the lower boundary temperature that determines cold weather concreting; and -5°C was used, as this is the lower temperature as determined by the scope of the project.

Throughout the curing period, dummy cylinders, with thermocouples set in the center of mass, recorded concrete temperatures every 30 minutes at each curing temperature. The temperatures were used to calculate the maturity index for both maturity functions. The maturity index is used with the results of the strength testing to develop a strength-maturity relationship.

Compression tests were performed at ages of 1 (23°C only), 3, 4, 7, 14 and 28 days (Fig. D1). Initial breaks for cylinders curing at -5 and 5°C occurred at 3-days, as it is estimated that for every 10°C decrease in temperature, the rate of strength gain may be half that of a mixture cured at room temperature. Cylinders cured at -5°C represents a very harsh curing condition. Despite this, the strength of this mixture exceeded 28 MPa at the end of the curing period. Cylinders cured at -5°C were warmed to approximately 5°C (in a 23°C room) prior to strength testing.

The strength gain rate constants, or K-values, for each curing temperature were determined from strength testing on 51-  $\times$  102-mm mortar cylinders. They were proportioned similar to the concrete mixture<sup>‡‡‡</sup> (w/c ratio of 0.39), and were cured, in their watertight cylinder molds, in water baths held at 23, 5 and  $-5^{\circ}$ C for up to 28 days. Flake calcium chloride, at a concentration of 23% by weight, was added to the water in the

Mortars were designed to simulate the mortar fraction of the concrete mixtures by calculating paste thickness on aggregate particles after all void spaces were theoretically filled.

 $-5^{\circ}$ C bath to keep it from freezing. The temperature of this coldroom hovered around the freezing temperature of the mixture, resulting in an average bath temperature of approximately  $-5.75^{\circ}$ C over the first 3 days of curing. Inspection of several samples revealed that the cylinders may have been damaged from this temperature being too close to the freezing point of the mixture and, consequently, they had not been gaining strength at an expected rate. Strength testing of the  $-5^{\circ}$ C mortar cylinders was suspended after 15 days. The  $-5^{\circ}$ C strength values, up to that point, still provided reasonable input to calculate the K-values because the data from the temperature profile of the dummy cylinders were similar to the profile of the compressive strength cylinders, that cured in air. As the strength cylinders used for the datum temperature cured in a water bath, this environment was more severe than curing in air. The initial break of the mortar cylinders (Fig. D2) was performed at an age of twice the final set time, shown the first data row of Table D1.

Table D1. Mortar compression tests in hours and times for corresponding curing temperature.

	23°C	5°C	–5°C
Test 1	10	40	75
Test 2	20	80	152
Test 3	40	160	225
Test 4	80	320	313
Test 5	160	640	362
Test 6	320	_	_
Test 7	640		

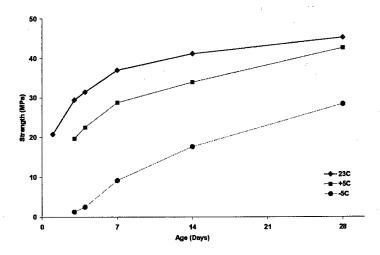


Figure D1. Average compressive strength of antifreeze concrete lab cylinders cured at 23, 5 and -5°C.

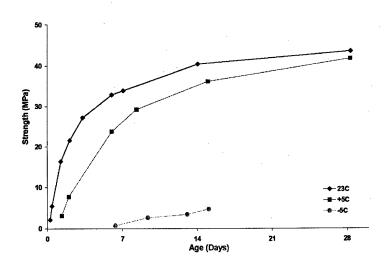


Figure D2. Average compressive strength of mortar cylinders used to determine the datum temperature and activation energy.

### **Results**

The reciprocal method was used to determine the K-values, in accordance with ASTM C 1074 (1998b). The values are listed in Table D2.

Table D2. Values used to determine K-values for each curing temperature.

Curing temperature (°C)	Final set time (hours)	Slope	Intercept value	R <sup>2</sup> (%)	<i>K</i> -value	Ln(K)
23	5	0.061	0.021	99	0.339	-1.08
5	20 <sup>†</sup>	0.106	0.020	100	0.189	-1.66
<b>–</b> 5	34	2.96 <sup>C</sup>	0.013	93	0.005	-5.41

<sup>\*</sup> Final set times on prepared mortars as determined from ASTM C 403 (1999b).

Determination of the datum temperature for the time-temperature method is shown in Figure D3. The datum temperature  $(-7^{\circ}\text{C})$  is the x-axis intercept, used throughout this study for the maturity analysis. In determining the datum temperature, strengths less than 4 MPa were not used, per ASTM C 1074 (1998b). The exception to this was the  $-5^{\circ}\text{C}$  strength data, which only utilized a total of three strengths in the analysis, two of which were below 4 MPa. Datum temperatures ranged between -2.5 and  $-18^{\circ}\text{C}$ , depending on

<sup>&</sup>lt;sup>†</sup> Laboratory set time at 5°C occurred between 13 and 24 hours; 20 hours was selected as approximately the average.

<sup>\*\*</sup> Using strength values from testing at 9, 13, and 15 days as these three points were closest to 4 MPa (ASTM C 1074 [1998b]).

which data points were analyzed. For the reciprocal method, using all of the data points resulted in a datum temperature of -2.5°C, which was higher than the design freezing point of the mixture. This temperature suggests that no strength gain will occur below this temperature. However, this is inconsistent with our lab results, as the antifreeze mixtures do gain strength at temperatures lower than this. A datum temperature of -7°C was obtained using the strength at ages of 160, 320, and 640 hours for the curing temperatures of 23 and 5°C, and with the -5°C data. For all three curing temperatures, the reciprocal values of the strength and age at these ages were the most similar. This datum temperature fell between the recommended values of -5 and -10°C, and provided reasonable strength estimations when used in the field. How critical is the datum temperature? It becomes apparent that using a temperature lower than -7°C, such as -18°C or less, increases the maturity index value and shifts the strength-maturity curve. For the time-temperature method, the maturity index is a rather artificial value. Although the selection of the datum temperature is somewhat arbitrary, it is important to use a consistent value. Until additional testing may be conducted, the reciprocal method is a relatively simple approach to determine the datum temperature, bearing in mind that a reasonable datum temperature may be the result of limiting the number of data points.

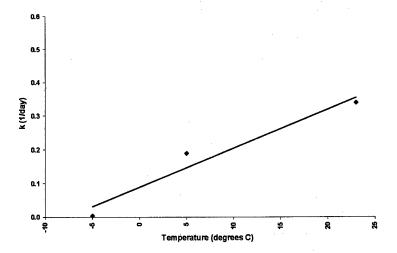


Figure D3. Strength gain rate constant values as a function of the curing temperature are used to determine the datum temperature (°C) following ASTM C 1074 (1998b).

The activation energy is needed for the equivalent age method. As shown in Figure D4, the natural log of the previously determined K-values is computed (Table D2) and plotted against the inverse of the absolute temperature. The  $R^2$  value of the best line fit was only 74%. The value of Q (11,319 K, which is the activation energy divided by the gas constant) is determined from Figure D4, following ASTM C 1074 (1998b), and re-

sults in an activation energy of 94,061 J/mol. This value is larger than suggested values of Q = 4700 K (Con-Cure Corporation). Using the Q value from the lab produced lower equivalent age values that underestimated the in place strength value. However, setting the Q value to 4700 K for the equivalent age calculations resulted in strength estimates with better correlation.

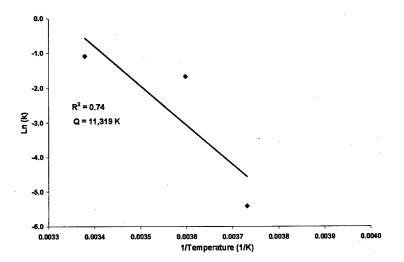


Figure D4. Determining the value of Q used for the equivalent age method (ASTM C 1074 [1998b]).

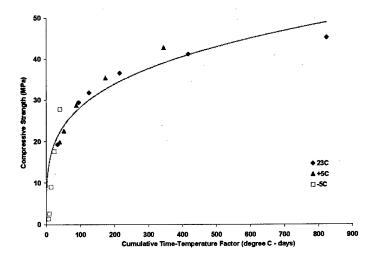


Figure D5. Actual compressive strength as a function of the cumulative time-temperature factor using lab-determined value of  $-7^{\circ}$ C as the datum temperature.

An indication that the maturity method could be used on antifreeze concrete is shown in Figures D5 and D6. Strength-maturity relationships for both methods were developed based on the 23°C curing temperatures. The time-temperature factor shows good correlation at early ages for the cylinders cured at lower temperatures.

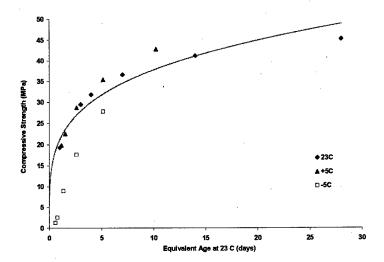


Figure D6. Actual compressive strength as a function of the equivalent age cured at 23°C, specified temperature  $(T_s)$ , and an activation energy (Q) of 4700 K.

Temperature and strength data were collected on the eight candidate antifreeze concrete mixtures while they were evaluated for low-temperature performance. Using this data, we developed strength-maturity curves for each mixture. The WRG II mixture is illustrated as an example in Figures D7 and D8. It became clear that the initial break at 23°C cure needs to begin earlier to better define the early age strength development, as the strength gain after 24 hours may exceed the target strength of 23 MPa. Earlier breaks, with the first occurring either 10–12 hours after the concrete was placed or a minimum strength of 4 MPa is reached worked best, with subsequent breaks occurring every 24 hours.

The strength-maturity relationships from Figures D7 and D8 are used to estimate the strength of test cylinders at 5 and -5°C (Fig. D9 and D10). The estimated strength, using the 23°C cure, is compared with actual strengths from compression cylinders cured at lower temperatures.

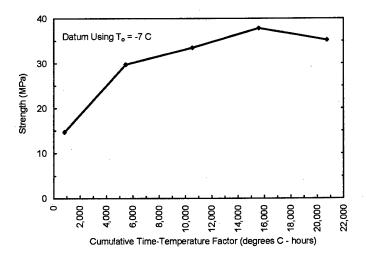


Figure D7. Strength-maturity relationship for time-temperature factor for WRG II mixture.

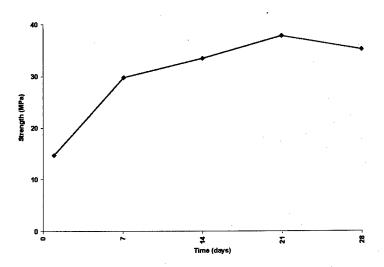


Figure D8. Strength-maturity relationship for equivalent age for WRG II mixture.

The estimated strength, using the time-temperature factor based on 23°C cure, worked relatively well for antifreeze concrete cured at 5°C, when compared to the actual strength (Fig. D9). For this mixture, the estimate made at 7 days exceeded the actual strength by 12% and then underestimates the strength, by less than 10%, for the remaining break ages. Using the time-temperature method for the other seven lab mixtures consistently underestimated the strength, with 20% being the largest difference between es-

timated and actual strength. At a curing temperature of 5°C, this method appears to give conservative strength values.

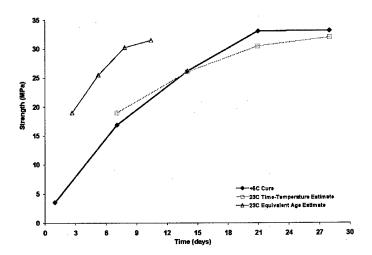


Figure D9. Strength estimation at 5°C for the WRG II mixture using both time-temperature factor and equivalent age methods compared to actual 5°C strength measurements.

Using the time-temperature function with temperature and strength data from the strength cylinders cured at 23°C to estimate the strength at -4°C (Fig. D10) tends to overestimate the strength. Of all eight mixtures tested, this mixture was the only one to overestimate the strength. The remaining seven mixtures all underestimated the strength value, by a wide range of 12-26% on day 14 and 21-31% on day 28. Because antifreeze concrete cures at a lower temperature, a strength-maturity relationship using 5°C data was developed, similar to using the 23°C, to see if this curing temperature estimated the strength more accurately. This did not result in a better strength approximation, with the exception of the WRG II mixture, which yielded the best results. For the other mixtures, using the 5°C curve resulted in a larger difference between the estimated and actual strength values, by as much as 46%. The equivalent age method overestimated the strength for all eight mixtures at both 5 and -4°C curing temperatures (Fig. D9 and D10). The specified temperature ( $T_s$ ) was adjusted to 5°C, but this did not improve the estimated strength.

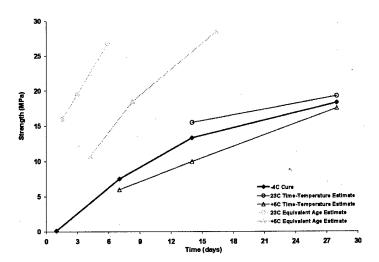


Figure D10. Strength estimation at -4°C for the WRG II mixture using both time-temperature factor and equivalent age methods compared to actual -4°C strength measurements.

A procedure similar to that used in the the lab was tested at each of the field demonstration sites. An experimental sidewalk (13 cm deep and 150 cm wide) was poured in Concord, New Hampshire. Prior to the pour, three instrumentation locations in the structure were selected. These locations should describe the structure thermally and spatially. Location 2 turned out to be the coldest place in this structure (Fig. D11). This was located along an edge roughly 30 mm below the surface and 130 mm in from the outside edge. This location was partially shaded during the day from a storage shed located not more than a few meters away.

In the field, strength cylinders are cast from the actual mixture used. This quality check is important because the actual mixtures used at the jobsite may be slightly different from the lab mixture. This, in fact, occurred at the field site in North Woodstock, New Hampshire, when the mixture had more water than the water content measurements indicated, most likely due to an overnight snowfall on the aggregate stockpiles.

Two curing regimes are recommended. One set cures at room temperature and the other in the field. The field cylinders cure in a picnic cooler in air. Similar to the lab, these cylinders cure under harsh conditions and are used to verify the strength gain at low temperatures, and they offer a "worst case" strength boundary, as the structure itself is unlikely to have achieved less strength gain than the field-cured cylinders. As discussed previously, these antifreeze mixtures can be sensitive, water content being a very important factor. Therefore, there is a real value in creating a strength-maturity curve in real-time with the actual mixture. This is illustrated in Figure D12 from the bridge curb pour

at West Lebanon, New Hampshire. The strength cylinders were broken at ages of 12 (23°C only), 23, 30, 36 (23°C only), 48, 74, 121, and 224 hours.

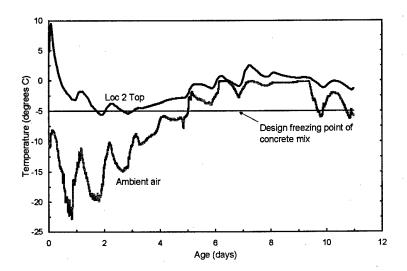


Figure D11. Temperatures at Concord, NH, field demonstration of ambient air and coldest instrumented location in concrete.

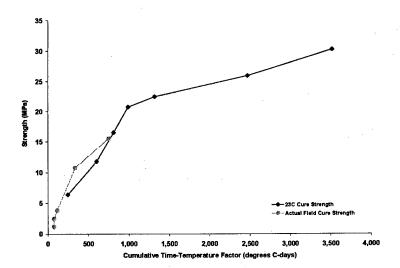


Figure D12. Two strength-maturity curves for the same concrete placed in Concord, NH—one using lab-cured samples and the other field-cured. Note the extra detail provided at lower strengths by the field-cured cylinders.

At the Concord field site, eighteen 76- × 152-mm plastic cylinder molds and two dummy temperature cylinders were cast and transported back to CRREL to cure at 23°C (no field-cured cylinders were cast at this site). The temperature readings and compressive strength measurements were used to develop the strength-maturity relationships shown in Figures D13 and D14. These strength-maturity relationships are then used to estimate the in-place strength of the structure (Fig. D15).

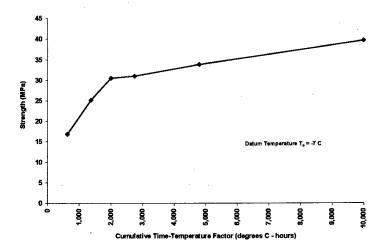


Figure D13. Strength-maturity relationship for Concord, NH, demonstration concrete using the time-temperature factor method.

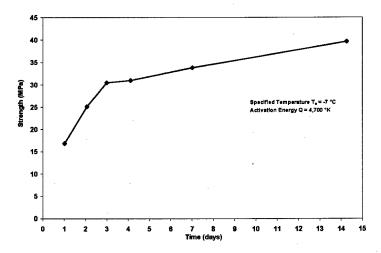


Figure D14. Strength-maturity relationship for Concord, NH, demonstration concrete using the equivalent age method.

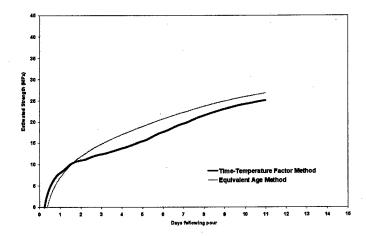


Figure D15. Strength-maturity relationships used to estimate in-place concrete strength development for Concord, NH, field site.

### **Conclusions**

Based on the data from the initial lab study and field demonstrations, the maturity method is appropriate for use with antifreeze concrete when the *concrete* temperature is 0°C and above. As shown in Figure D6, the equivalent age maturity calculations at -5°C do not correlate as well as for the early age section of the curve as the time-temperature factor (Fig. D5). Applying conventional practices to the antifreeze concrete revealed that both methods lose the ability to estimate the in-place strength when the concrete temperatures remain below zero for a significant length of time. While this finding may become more of an issue when emplacing antifreeze concrete at even lower temperatures than the scope of this project, this is not viewed as a significant problem. The temperatures from the Concord site (Fig. D11) illustrate that even when the air temperature drops well below zero, the concrete temperature did not reach -5°C until approximately 2 days after the concrete was placed. This allowed the concrete time to gain early age strength and use up water for cement hydration, which in turn offers continued protection against freezing. In the field, we do not anticipate extended low temperatures similar to those we used in the initial lab study.

In general, basing the strength-maturity relationship on room temperature results, as called for in ASTM C 1074 (1998b), results in good strength estimation when the concrete temperatures are 0°C and above. The time-temperature factor approach resulted in more conservative strength estimates, as it tended to underestimate the actual strength of the structure. The equivalent age method tended to overestimate the strength and requires more study, as clarification is needed for the calculation constants.

The strength-maturity relationships created under controlled conditions in the lab provide an initial guideline of how a mixture will gain strength in the field. At the field site, the components of the mixture are likely to be slightly different (i.e., w/c ratios, admixture dosage, etc.). Therefore, it is recommended that strength cylinders be cast from concrete actually placed for the job to confirm the strength gain relationship most accurately. Two curing regimes are recommended, room temperature and field-cure, when predictions at early age are desired. Initial room temperature breaks of antifreeze concrete 24 hours old regularly produce strengths near or greater than 20 MPa. Breaking standard cured test cylinders as early as 8 hours or as close to 4 MPa is recommended. Field-cured cylinders, because they develop strength gradually in the cold, can also provide additional data at low maturities, but allow breaks to occur at later ages. Low-strength results are better in defining the beginning of the strength curve and, in turn, this section allows better estimates of the lower strengths associated with cold curing temperatures. The time of the first break is critical as it should be early enough that a strength of at least 4 MPa is attained, as specified in ASTM C-1074.

The Concord field site (Fig. D15) showed that the estimated strength of the coldest location in the sidewalk reached 20 MPa in approximately 7.25 days using the time-temperature factor, and roughly 5.75 days using the equivalent age method. With such low air temperatures, the forms were removed 10 days after the pour and construction operations continued. The use of the maturity method allowed the strength estimation to happen quickly so construction operations could continue on schedule.